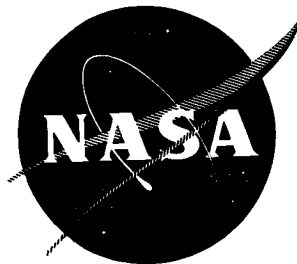


N71-23167

NASA CR-72380



OXYGEN DIFLUORIDE RESEARCH STUDY

by

**CASE FILE
COPY**

Ralph B. Jackson
ALLIED CHEMICAL CORPORATION

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
CONTRACT NAS 3-2564
Theodore Male, Project Manager

NOTICE

This report was prepared as an account of Government-sponsored work. Neither the United States, nor the National Aeronautics and Space Administration (NASA), nor any person acting on behalf of NASA:

- A.) Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately-owned rights; or
- B.) Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method or process disclosed in this report.

As used above, "person acting on behalf of NASA" includes any employee or contractor of NASA, or employee of such contractor, to the extent that such employee or contractor of NASA, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with NASA, or his employment with such contractor.

Requests for copies of this report should be referred to

National Aeronautics and Space Administration
Scientific and Technical Information Facility
P. O. Box 33
College Park, Maryland 20740

FINAL REPORT

OXYGEN DIFLUORIDE RESEARCH STUDY

by

Ralph B. Jackson
ALLIED CHEMICAL CORPORATION
Morristown, New Jersey 07960

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

December 1970

CONTRACT NAS 3-2564

NASA Lewis Research Center
Cleveland, Ohio
Theodore Male, Project Manager
Chemical Rocket Division

FOREWORD

This report was prepared by Allied Chemical Corporation under NASA Contract No. NAS 3-2564. The program was initiated and administered by Lewis Research Center, Chemical Rocket Division. The Project Manager for the contract was Mr. Theodore Male.

This report has been prepared in two volumes. This Volume covers the laboratory investigation conducted between June 1963 and September 1965. The other volume is an oxygen difluoride handling manual which includes information available to December 1967. The work was performed by the Industrial Chemicals Division of Allied Chemical Corporation at Morristown, New Jersey, and the report was prepared by Mr. R. B. Jackson. Acknowledgement is given to Mr. J. M. Siegmund for his significant contributions and assistance to the author throughout this entire investigation.

TABLE OF CONTENTS

<u>Section</u>		<u>Page</u>
1.	Introduction.....	1
2.	OF ₂ Spillage Control.....	3
2.1	Gas Phase OF ₂ Decontamination Study.....	3
2.2	Liquid Phase OF ₂ Decontamination.....	8
2.3	Liquid OF ₂ Spill and Deluge.....	13
3.	Material Compatibility.....	21
3.1	Preliminary OF ₂ Exposure.....	21
3.2	Twenty-Four Hour Gaseous OF ₂ Tests.....	25
3.3	Seven Day Gaseous OF ₂ Tests - Procedure.....	26
3.4	Liquid OF ₂ Storage Tests.....	27
3.5	Tensile Tests.....	33
4.	Dynamic Testing of Materials in Liquid OF ₂ ...	41
4.1	Apparatus and Equipment.....	41
4.2	Procedure.....	42
4.3	Orifice Calibration.....	43
4.4	Test Procedure - Test #1.....	44
4.5	Test Procedure - Test #2.....	47
4.6	Explosion Report.....	51
5.	Investigation of Explosive Burnout.....	61
5.1	Apparatus and Equipment.....	61
5.2	Test Procedure.....	68
5.3	Experimental Data.....	70
5.4	Conclusions.....	76
6.	References.....	88

LIST OF TABLES

<u>Number</u>		<u>Page</u>
1	OF ₂ Decontamination Study - Gas Phase Tests.....	89
2	OF ₂ Decontaminants.....	91
3	OF ₂ Decontaminants - Preliminary Economic Eval...	92
4	OF ₂ Decontamination Study - Liquid Phase Tests...	93
5	OF ₂ Decontamination Study - Spray Chamber Tests..	94
6	OF ₂ Decontaminants - Final Economic Evaluation...	98
7	Candidate Materials - OF ₂ Compatibility Tests....	99
8	Material Compatibility in OF ₂ - Preliminary Screening.....	102
9	Material Compatibility - 24 Hour OF ₂ Gas Exposure.....	104
10	Material Compatibility - 7 Day Gaseous OF ₂ Exposure.....	106
11	Material Compatibility - 48 Hour Liquid OF ₂ Exposure.....	108
12	Material Compatibility - 7 Day Liquid OF ₂ Exposure.....	110
13	Material Compatibility - Tensile Tests.....	112
14	Material Compatibility - Crystallinity.....	119
15	Orifice Calibration - CTFE Orifice 0.0135" Dia...	120
16	Liquid OF ₂ - Dynamic Test #1.....	121
17	Annin Valve - Component Identification.....	122
18	Annin Valve #1 - Component Weight Losses and Measurements.....	123
19	OF ₂ Burnout Investigation - Preliminary Tests Summary.....	124
20	OF ₂ Burnout Investigation - Final Tests Summary..	125

LIST OF FIGURES

<u>Number</u>		<u>Page</u>
1	OF ₂ Gas Phase Decontamination.....	126
2	Liquid OF ₂ Decontamination Study.....	127
3	OF ₂ Decontamination Spray Chamber.....	128
4	Storage Tests OF ₂ Bomb.....	129
5	OF ₂ Compatibility Study - Gas Phase Tests.....	130
6	OF ₂ Compatibility Study - Liquid Phase Tests....	131
7	Charcoal Burner.....	132
8	Instron Cryogenic Adapter.....	133
9	Liquid OF ₂ Dynamic Test Setup.....	134
10	Specimen Holder - OF ₂ Dynamic Test.....	135
11	Annin Valve.....	136
12	OF ₂ Burnout Study.....	137
13	OF ₂ Burnout, Investigation, Reactor.....	138
14	OF ₂ Burnout Investigation, Electrode Adapter....	139

APPENDIX

Exhibits

A - W OF₂ Explosion Photographs

ABSTRACT

Decontaminants for the control of oxygen difluoride (OF_2) spills were evaluated. Dilute aqueous solutions of ammonia were found to be the most effective decontaminants.

The compatibility of 40 plastic and elastomeric materials with gaseous and liquid OF_2 under static conditions was investigated. The most resistant materials were found to be tetrafluoroethylene, chlorotrifluoroethylene, and fluorinated ethylene-propylene polymers.

The materials found to be most compatible in the static evaluation were selected for testing under dynamic OF_2 conditions. This program was interrupted during the second test when an explosion destroyed the test equipment.

An investigation of the phenomenon of "explosive burnout", was conducted. Instant and complete decomposition of OF_2 gas, initiated by thermal shock, failed to produce detonation or burnout.

INTRODUCTION

Oxygen difluoride (OF_2) is a highly energetic, space storable oxidizer which possesses great merit as a rocket propellant. To utilize its great potential it is first necessary to acquire the knowledge that will permit it to be used effectively and safely.

Past experience has shown that spills of varying magnitudes will occur during rocket engine development. It is essential that hazards and damage be kept minimal. Therefore, adequate means to control spills and decontaminate test areas must be developed.

From the standpoint of hardware design it is not desirable to be limited to the use of metals for such critical components as shaft seals, gaskets, valve seats, and the like. Design problems can be greatly simplified if elastomers or plastics can be used. The compatibility of such materials with liquid OF_2 under both static and dynamic conditions must be established.

Incidents have been reported in which OF_2 has unexpectedly reacted quite vigorously with associated hardware, resulting in the burnout of valves, lines, and sundry components at several points in the system. The rapidity with which this phenomenon occurs has led to its description as "explosive burnout". An understanding of the initiating mechanism for this phenomenon is necessary.

It is also desirable that a manual be available as a reference for all phases of OF_2 handling. All pertinent available information should be incorporated into this manual so that it can serve as a complete and convenient guide for OF_2 handling procedures.

This research study is directed toward furnishing this necessary information. It consists of five separate tasks: spillage control, static material compatibility evaluations, dynamic testing, burnout investigation, and the compilation of an OF₂ handling manual. The first four tasks, which involve laboratory experimentation, have been included in this report. For greater utility and convenience, the handling manual appears as a separate volume.

2. OF₂ SPILLAGE CONTROL

The purpose of this phase of the program was to develop adequate means to control or decontaminate spills involving liquid and vapor oxygen difluoride (OF₂). This material, in addition to being an energetic oxidizer, is considered to be quite toxic. It is therefore imperative that suitable materials and methods of application be established to decontaminate such spills. A decontaminant to be suitable must meet several criteria. It must react smoothly, readily and relatively completely. It must in itself produce no additional hazards by nature of flammability, toxicity or corrosivity. In addition, the reaction by-products must not present any undue hazards. Lastly, the decontaminant should be readily available and economically feasible. Our investigations indicated that a dilute aqueous solution of ammonia met all the preceding criteria and can therefore be recommended as a suitable OF₂ spill decontaminant.

2.1. Gas Phase OF₂ Decontamination Study

This investigation was the first of a series of three tests designed to evaluate the performance of candidate decontaminants with OF₂. This initial effort was designed to neutralize OF₂ gas and was meant to screen out materials that were either ineffective as neutralizing agents or materials whose reactions were too vigorous. Approximately 50 test runs were made in which 26 decontaminant solutions were evaluated. This investigation showed that dilute aqueous solutions of ammonia (NH₄OH) were the most effective decontaminants for OF₂ gas.

2.1.1.

Apparatus and Equipment

The handling of OF_2 requires the same caution and safeguards as are used for fluorine. Therefore the OF_2 cylinder was secured in a steel enclosure and all valve handles were extended through this enclosure. The main cylinder valve was operated from outside the laboratory by means of a system of rods and gears which extended through the laboratory wall.

The apparatus and equipment used in this study is shown schematically in Figure 1.

Flow control was maintained through two Hoke M343 needle valves in series. The OF_2 was also passed through a NaF pellet packed unit to scrub out any possible residual traces of HF in the gas. An elaborate manifold system had been set-up so that all lines could be used to carry He, OF_2 , or N_2 , at any rate, as desired. All lines were 1/4" copper tubing with Swagelok fittings.

Fischer-Porter tri-flat rotameters were used for metering the OF_2 . It should be noted that considerable effort was required to obtain accurate calibration of the meters. Potassium iodide reaction with OF_2 and the subsequent titration of the liberated iodine was found to be slightly inaccurate. Positive gas displacement techniques were eventually developed which provided the desired accuracy.

The heart of the system was two Hoke solenoid valves, one normally opened and one normally closed. The first (No. 1) was in our vent line while the other (No. 2) controlled the flow to our decontamination set-up. The OF_2 flow through the rotameter was adjusted before the two valves were simultaneously activated by a single switch. The switch remained open for a predetermined time, and the amount of OF_2 that was introduced into the candidate decontaminant was extremely accurate.

The test solution was then analyzed for fluorine pickup by standard colorimetric determinations. All tests were run in duplicate. As a check, the fluoride content of the unexposed decontaminant solution was determined to further assure the accuracy of the results.

The disposal of OF_2 was accomplished by introducing the waste gas into the luminous flame of a bunsen burner where it was completely neutralized. No traces of OF_2 could be detected at the top of the stack using the standard KI test. This method could no doubt be adapted to large scale OF_2 waste disposal.

2.1.2. Experimental Procedure

This program considered the reactions between approximately 0.05 grams of OF_2 gas and 50 cc. samples of aqueous solutions of the candidate decontaminant. The gas was bubbled through the test liquid at a carefully controlled rate. Extreme care was taken to duplicate all the test conditions for each run. Visual observations were made

of each reaction. The spent solution was then analyzed for fluoride pickup. The fluoride recovery was used to compute the percentage of OF_2 decontaminated or neutralized. This technique did not produce exact quantitative data since some gaseous by-products escaped as did, to some slight degree, the by-products of the reaction in the vapors above the test solutions. However, analysis of the vent gases which were scrubbed with water showed nil, to insignificant, fluoride pickup. The technique as described therefore appeared to be quite satisfactory since all test materials were exposed under identical conditions and good comparative data was obtained.

2.1.3.

Experimental Data and Conclusions

The data from fifty-two separate runs covering twenty-six materials are shown in Table 1. The milligrams of fluorine shown in this table are the amounts found in the test solutions. This was used to calculate the percent OF_2 decontaminated. The density of OF_2 gas used in these calculations was 2.41 mg/cc.

The most suitable decontaminants on the basis of this study were dilute aqueous solutions of ammonia. A 5% solution (NH_4OH basis) decontaminated over 81% of the OF_2 . To demonstrate that the concentration of such a decontaminant was not critical, relative to violence of reaction, 7-1/2% and 10% solutions were used in similar tests. The 7-1/2% solution showed no appreciable improvement while the 10% solution captured approximately 90% of the OF_2 .

Considering that a 5% solution is relatively innocuous to personnel, but still effective as a decontaminant, it is felt that this strength is to be preferred in any large scale decontamination set-up. However, these tests indicated that stronger concentrations may be safely used.

Only four other materials accomplished fifty percent decontamination. None of these, from a practical standpoint, are as suitable as the ammonia solutions. In fact, the reaction of one material, isopropylamine, which showed an average decontamination of 53%, was accompanied by flashes and mild, but audible, explosions. This pyrotechnic display alone would remove isopropylamine from further consideration.

2.1.4.

Economic Evaluations

The materials used in this program were generally of reagent or high purity grade. This was done to assure the purity of the test solutions, thus eliminating side reactions due to possible impurities. A list of the actual materials used is shown in Table 2.

In actual field decontamination, however, technical grade materials would be preferred simply on an economical basis. Table 3 shows the economics of the various decontaminants. It should be noted that a 50:1 decontaminant to OF_2 ratio was used in these calculations, since this was the approximate ratio used in the gas phase decontamination test series. Therefore, one ton of decontaminant

would react with forty pounds of OF_2 . The approximate effectiveness, which was obtained from the % OF_2 decontaminated, was then used to get an accurate relative cost. The estimated costs to neutralize one hundred pounds of OF_2 therefore are comparative figures. This comparison will hold even if the decontaminant to OF_2 ratio is reduced. No doubt in a large scale decontamination set-up the ratio would be considerably lower. The estimated cost per ton is based on 100% material* at the lowest available price. In all cases, the water costs are equal (all solutions are 95% H_2O) and are therefore not considered a factor in this economic estimate. Again, weighing all these factors, dilute aqueous solutions of ammonia appear to be by far the most economic decontaminants.

2.2.

Liquid Phase OF_2 Decontamination

A second series of tests was run involving liquid OF_2 and aqueous solutions of those candidate decontaminants which had shown some significant merit in the previous gas phase tests. In addition several dry powders were evaluated with liquid OF_2 . This series had as its primary purpose the determination of the compatibility of the decontaminant with liquid OF_2 . It was also hoped that some significant quantitative data relative to the effectiveness of the test materials could be obtained. Due to the nature of these experiments, little information was obtained on the comparative efficiencies of the decontaminants.

*Candidate decontaminant excluding water

This series did emphasize that liquid OF_2 does not react extremely violently with many substances and verified that dilute aqueous ammonia solutions are safe decontaminants.

2.2.1. Apparatus and Equipment

The OF_2 system used for the gas phase OF_2 decontamination tests was slightly modified for this series of tests. The feed system to the OF_2 condenser or receiver was set-up to flush the receiver with helium before OF_2 was introduced. A helium purge was maintained in the upper section of the receiver during OF_2 condensation to prevent the entrance and condensation of air. A representation of this set-up is shown in Figure 2. The condensed OF_2 was a slightly cloudy yellow liquid. The cloudiness was possibly an indication of the presence of CO_2 crystals since the OF_2 used in this work was taken directly from the cylinder with no additional purification.

When the desired amount of OF_2 had been condensed, the Dewar containing the LN_2 was removed and the beaker containing the test solution was put in place. The calibrated receiver was then broken by means of a spring loaded plunger which was triggered from behind a barricade. The liquid OF_2 then spilled into the beaker containing the test material.

2.2.2.

Experimental Procedure

In this series of tests known volumes of OF_2 were condensed in calibrated receivers which were cooled with liquid nitrogen. The Dewar flask containing the liquid nitrogen was then removed and a beaker containing a measured volume of test decontaminant was placed under the OF_2 receiver. In rapid order, the receiver was broken allowing the liquid OF_2 to pour into the test solution. After all the OF_2 had boiled off, the residual material was analyzed for fluoride pickup. The same standard analytical procedure was used as was used in the previous series of tests.

It was noted that the liquid OF_2 generally sank to the bottom of the beaker and formed one or more beads or droplets of OF_2 . These OF_2 globules rose to the surface and fell back to the bottom repeatedly in a "YO-YO" like motion. It was felt that if any reaction occurred between the OF_2 and the test material, it took place at the surface of the solution rather than when the OF_2 was submerged. The analytical data showed extremely poor recovery in the liquid. The vapors above the liquid however showed in some cases copious fuming indicating vapor phase reaction. This was especially true when the ammonia solutions were involved. However, the physical set-up was such that monitoring the off gases was not feasible and quantitative decontamination data was not obtained in this series.

Experimental Data

The data from the twenty-one runs are shown in Table 4. It should be noted that in addition to aqueous solutions, three dry powders were also investigated in this series of tests. These powders had shown some merit as decontaminants for fluorine in a previous program (Ref. 1). However, in these tests conducted under similar conditions, they were ineffective when used for liquid OF_2 spill control.

The several test liquids all showed low fluoride pickup. Since visual observations indicated little or no reaction between the liquid, this result was not surprising. The reactions appeared to occur at or above the surface of the solutions and the gaseous by-products thereby escaped to the atmosphere. It was conjectured that the liquid OF_2 may actually have formed a thin shell of ice or OF_2 gas around the globule. This "shell" caused the droplets to rise to the surface where some OF_2 gas escaped. The remaining oxidizer then fell back through the liquid. The repetition of this cycle until the OF_2 was depleted explains the previously described "YO-YO" effect.

Further evidence that reaction occurred above the surface was the formation of white fumes above the liquid. The ammoniacal solutions showed copious fuming. Since there is an appreciable amount of NH_3 in the vapor above the liquid, this fuming was indicative of a high degree of gas phase reaction.

To further establish the compatibility of aqueous ammonia solutions and liquid OF_2 , we made several runs wherein we increased the amount of liquid OF_2 and decreased the volume of test solution. The final ammonia run involved 5 ml. or 9 grams of liquid OF_2 in 100 ml. of 5% NH_4OH . Again, the two appeared to be completely compatible.

2.2.4.

Conclusions

Based on the previously established criteria for determining a suitable decontaminant, we can recommend dilute aqueous solutions of ammonia. Of paramount importance, it proved to be the most effective material tested. In addition, dilute ammonia is relatively non-hazardous. It is neither flammable nor toxic. In addition, the by-products are considered relatively non-toxic. Lastly, it is not only plentiful, readily available, but as shown in Table 3, it is economically feasible.

2.3. Liquid OF₂ Spill and Deluge

The final evaluation of candidate decontaminants were based on results obtained from a spray chamber test. This test was designed to simulate an actual liquid OF₂ spill followed by a spray deluge of test solutions. Since the apparatus permitted the recovery of the spent spray and reaction by-products, it provided a means of measuring the comparative effectiveness of the several decontaminants.

2.3.1. Experimental Procedure

A spray chamber had been designed and fabricated (Figure 3) to simulate a spill of liquid OF₂ followed by a spray of a candidate decontaminant solution. A measured amount of OF₂ (4.5 gms) was condensed in a test tube mounted inside the chamber. The test tube was then broken by a remote controlled triggering mechanism to spill the OF₂. Simultaneously, a solenoid valve was energized thus permitting the test solution to deluge the spill. The collected spent liquor was analyzed for fluoride content and the % OF₂ neutralization was then calculated. All tests were run in duplicate.

2.3.2. Apparatus and Equipment

The test tubes used in this study had been carefully calibrated. In a typical run, the test tube was slipped over the copper cold finger and held in place with two clamps. To assure breakage when the trigger mechanism was released, an "anvil" was backed up snugly against the side of the tube. After the chamber window was secured in place,

the chamber was purged with nitrogen to prevent frosting on either the test tube or cold finger when the LN_2 flow was started. The OF_2 line and test tube were purged with helium both before and during the filling of the cold finger with LN_2 . The OF_2 flow was then started and the gas condensed in the tube until the desired liquid level was obtained. Excess OF_2 in the lines was removed with helium. With the required amount of OF_2 condensed in the tube, in rapid order, the nitrogen purge was shut off, the trigger was pulled breaking the test tube, and the spray solenoid valve was activated. The decontaminant solution had previously been charged to the spray reservoir and the system pressurized. The pressure had been preselected to give the desired flow rate in ml./min. of decontaminant spray. The decontaminant solution to OF_2 ratio was controlled by timing the spray duration with a stop watch.

After decontaminating the spilled OF_2 , the chamber was allowed to drain for 15 minutes. Nitrogen was slowly purged into the chamber to remove any unreacted OF_2 and uncaptured by-product gases, and the drain was opened to collect the spent liquid. The volume of liquid was measured as a check against a possible malfunction of the solenoid valve and the spray nozzle.

The nozzles used initially in this test were Spraying Systems Co. No. 5500X-1 which produced a full cone jet. However, this nozzle was available only in brass. Brass is not satisfactorily resistant to aqueous ammonia solutions and this caused frequent re-calibration and replacement.

We therefore switched to a stainless steel nozzle, Spraying Systems Co. No. 1/8 G.G.S.S.-1 Fulljet nozzle. This nozzle was as similar to the 5500X-1 as we could obtain. The cone shape and the pattern of the spray were quite similar. However, the orifice was larger and higher rates and spray ratios were used with this nozzle.

The collected spent spray, and in a few test runs, spray chamber rinse waters were analyzed for fluoride content by standard techniques. The fluoride content was then used to calculate the amount of OF_2 that had been captured.

2.3.3. Experimental Data

The results of this extensive spray chamber test program are shown in Table 5 which covers 50 tests. Several other runs which were aborted or spoiled due to mechanical malfunctions were discarded. The initial tests, in which straight water was used, showed the expected low OF_2 neutralization. However, a water run (SN 2A) which showed a 30% decontamination was accompanied by a bright flash when the test tube broke. It should be noted that, in these initial tests, the chamber was inadequately purged of moisture during the cooldown and OF_2 condensation steps. As a result a moderate deposit of rime had formed on the outside wall of the test tube. It is believed that the observed flash was indicative of a reaction between the ice and the OF_2 . We had previously demonstrated that

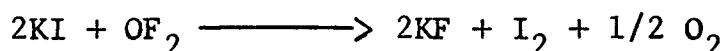
normally neither liquid F_2 or OF_2 react vigorously with water or ice. However, investigations by Astropower, Inc. (Ref. 2) showed that such mixtures are impact sensitive. Therefore, the flash we observed suggested that the tube breaker supplied sufficient energy to initiate the OF_2 -frost reaction.

Since the preliminary gas phase OF_2 tests had shown that dilute NH_4OH solutions were the best decontaminants, extensive testing was done with this material. Twenty-seven runs were made with a 5% aqueous solution of NH_4OH . Spray to oxidizer ratios ranged from 15:1 to 90:1 with spray rates from 50 to 750 ml./min. Since the lower ratios did not contain the stoichiometric quantities of NH_4OH necessary to completely neutralize the OF_2 (4.5 gms/run), the results were low as was expected. The ammoniacal tests were conducted using a brass spray nozzle. As noted previously the resultant corrosion occasionally caused fluctuations in the spray rates. This can be readily seen in Table 5 wherein the collected liquor in some runs exceeded the calculated delivered spray. At the low spray ratio the percent OF_2 decontaminated or neutralized was far below the 82% neutralization figure obtained in the gas phase tests performed previously. However, when the ratio of spray to OF_2 was increased to 90:1, OF_2 neutralization approached 50%. It should be noted that the active decontaminant constitutes only 5% of the spray, the water alone having negligible effect. In previous decontamination studies on F_2 (Ref. 1) and ClF_3 (Ref. 3), the water per se also acted as a decontaminant. Therefore, lower spray ratios

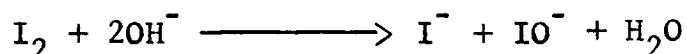
were used with high effectiveness. OF_2 does not readily hydrolyze, and is in fact slightly soluble in water. Therefore, the 90:1 spray ratio provided a decontaminant to OF_2 ratio of 4.5:1. This is far less than the decontaminant ratio of 50:1 used in the previous gas phase tests.

After reaching the 90:1 ratio, we changed to a stainless steel nozzle to alleviate the constant problem of nozzle recalibration and replacement. The larger orifice in the stainless nozzle required a tenfold increase in spray rate to obtain a similar spray pattern. This reduced the contact time and the spray therefore was somewhat less effective. We noted that the short-spray period resulted in a much more dense cloud of NH_4F remaining in the test chamber after the run and heavier fumes in the exit. We therefore made some runs (14A & C) followed by a light rinse of the tower. The water rinse, which removed fluorides from the chamber walls, picked up about half as much fluoride as was recovered in the spent liquor. In one run the total fluoride from run and rinse was about 45%. This total recovery compared well with capture for runs at the same spray ratio with low spray rates. It was therefore assumed that most of the reaction occurred early in the spray period, but the by-product fumes were largely knocked down and captured in the latter portion of the spray period. Run 12 was made to verify this theory and the spent liquor was collected at intervals while the spray continued. The results though not as conclusive as we would like, do tend to give credence to this theory.

Following the 5% NH_4OH tests, several other decontaminants were tried, only two of which showed significant OF_2 decontamination. These materials, 5% KI in 1% KOH solution and 5% Na_2SO_3 in 1% NaOH solution, showed about 59 and 48% neutralization respectively. The reaction with the KI solution is believed to proceed as follows:



In basic solutions I_2 disproportionates to form iodide and hypoiodite ions:



The by-products of the KI decontaminations therefore can not be considered unduly hazardous.

The sodium sulfite reaction is basically an oxidation-reduction mechanism, forming sulfates and fluorides. Here too, no unduly hazardous by-products are formed.

2.3.4.

Conclusions

From the results of these tests it can be seen that three materials show relatively high effectiveness as decontaminants. These materials are:

1. 5% NH_4OH solution
2. 5% Na_2SO_3 in 1% NaOH solution
3. 5% KI in 1% KOH solution

All three materials can be handled without undue hazard. The by-products of the reaction with OF_2 are relatively non-toxic. However, on an economic basis (Table 6) the NH_4OH is far superior. It would cost less than half as much as the Na_2SO_3 in caustic. The KI solution by comparison is simply economically not feasible.

A second and possibly even stronger reason for preferring ammonia solutions is the fact that decontamination occurred in both the liquid and vapor phases. This was indicated by the white NH_4F fumes in the vent exit and the significant deposit of fluorides remaining on the chamber walls after the spray was shut off. The other two decontaminants apparently reacted only in the liquid phase creating a hit-or-miss situation. The full capacity of the ammonia was not determined since no efforts were made to trap the escaping fumes. It should be noted that this would also be the case in a real spill. However, the escaping gases are to a large extent neutralized. In the sulfite and iodide tests most of the unrecovered OF_2 could be assumed to have escaped as unreacted, toxic OF_2 . It should be noticed that all runs conducted with these materials proceeded smoothly and quickly. Use of these decontaminants therefore does not present any problem from a safety standpoint.

From a practical standpoint the use of NH_4OH presents the least problem. It can be stored as NH_3 in cylinders

and fed into the water line feeding the spray heads upon activating the spray. Therefore, no large tanks of NH_4OH solution are needed. The Na_2SO_3 and caustic on the other hand must be properly dissolved in water and stored in tanks. This, of course, limits the amount of sulfite that can be made available. The ammonia deluge on the other hand can be continued as long as the supply of manifolded cylinders lasts and water remains available. The ammonia thus stored is extremely stable. The sulfite in solution on the other hand will be slowly oxidized to ineffectual sulfate by the oxygen dissolved in the water or by the air in the tank ullage.

Based on the cited results and conclusions, we therefore strongly recommend spray deluges containing dilute aqueous solutions of ammonia as a suitable means of decontaminating or neutralizing spills of liquid oxygen difluoride.

3.

MATERIAL COMPATIBILITY

This phase of the program concerned the evaluation of elastomeric and plastic materials for compatibility with liquid oxygen difluoride (OF_2). The list of candidate materials was compiled from a variety of sources ranging from actual service experience to commercial advertisements. This broad gamut was chosen primarily to refute or confirm the elaborate claims of various vendors as well as to ascertain the merits of materials presently used in OF_2 service. In addition, many materials suggested or recommended by others for consideration in this study had been included.

Although we were well aware that many materials would prove worthless, we felt that the elimination of such materials from future consideration could prevent failures or accidents. Manufacturers of the candidate materials were advised of the conditions to which we intended to expose their products. Several admitted that, despite their published claims to the contrary, their materials would be unsuitable. Others, perhaps overly optimistic, desired to have their products included. A complete list of the materials therefore considered for this program may be found in Table 7.

3.1

Preliminary OF_2 Exposure

A series of four preliminary tests were performed on the candidate materials to eliminate any that would readily react with OF_2 . In all such tests, a small piece of material was used so that if any violent reaction occurred, the damage would be minimal. No attempts were made to obtain quantitative data from these preliminary tests which are described in the following sections.

3.1.1. Test 1 - Gaseous OF₂ Exposure

The specimens were thoroughly washed and rinsed with distilled water and then completely dried in a stream of dry nitrogen. Solvent washing was avoided since the compatibility of many of the materials with organic solvents was unknown. The cleaned samples were thereafter handled with tweezers to avoid contamination and stored in marked polyethylene bags before testing.

The cleaned specimen was placed in a clean pyrex trap from which dry nitrogen was displaced by OF₂. After a complete OF₂ atmosphere was obtained, a slow flow of OF₂ was passed through the trap for fifteen to twenty minutes. The system was then flushed with dry nitrogen and the sample stored for further testing in liquid nitrogen. The trap was protected with a plexiglass shield which also facilitated visual inspection during the exposure period. To avoid any contamination, only one specimen was tested at a time.

3.1.2. Test 2 - Liquid Nitrogen Exposure

In an effort to obtain a preliminary evaluation of the candidate materials' suitability for cryogenic service, samples were exposed to liquid nitrogen. The samples used in Test 1 (gaseous OF₂) were immersed in liquid nitrogen for several minutes after equilibrium was obtained. An unsilvered dewar was used to contain the LN₂ so that visual observations could be made for physical changes such as spalling or cracking.

After exposure, the specimens were quickly removed, and dropped a distance of one foot onto an alberene stone laboratory work bench. Specimens were then examined for cracking, chipping, or other signs of embrittlement. Upon warming to room temperature, specimens were given a rough check to see if they had regained their former flexibility.

3.1.3. Test 3 - Liquid OF₂ Exposure, Cooled Specimens

The specimens that passed the first two tests were then exposed to liquid OF₂. Each specimen was placed in a clean test tube which was immersed in liquid nitrogen. The OF₂ gas was then introduced into the test tube and condensed until the liquid OF₂ covered at least three quarters of the specimen. The specimen remained thus immersed for fifteen to twenty minutes. The liquid nitrogen was then removed and the OF₂ allowed to slowly evaporate. As in Test 1, continuous visual observation of the specimen was maintained. After all the OF₂ had evaporated and the tube was flushed with nitrogen, the specimen was removed and examined for any signs of degradation or reaction. Again, only one specimen was tested in each run.

3.1.4. Test 4 - Liquid OF₂ Exposure, Uncooled Specimens

In Test 3, the specimens were pre-cooled before exposure to OF₂. It was felt that the cold specimens were less reactive than warm specimens. In this test, the effect of exposing room temperature specimens to liquid OF₂ was determined. This test was thought to more closely approximate

certain service conditions wherein warm parts are suddenly chilled. Examples of this would be the initial filling with OF_2 , or the initiation of certain dynamic operations. The OF_2 was again condensed as described in Test 3. The specimen however was suspended above the test tube during OF_2 filling and remained at ambient temperature. When the required liquid OF_2 level was obtained, the specimen was released, falling into the OF_2 . It remained in the liquid for an additional fifteen to twenty minutes. The OF_2 was then evaporated and the specimen examined after purging with nitrogen. Again, when possible, the identical specimens were used as had been used in the previous tests.

3.1.5. Results

Approximately 40 materials were screened in these tests. Of the materials tested, eleven failed to survive this preliminary evaluation and were excluded from the more extensive testing phases of this investigation. A list of the materials tested and the results are shown in Table 8. The main cause for elimination, described as surface degradation, covered such factors as surface cracking and discoloration. In the case of the silicone rubbers that were thus eliminated, the surface cracking was often not observed until several hours after exposure. A rating of "unchanged" indicated that the specimens showed no significant signs of reaction or degradation.

One material, Capran 77C nylon film, was re-tested. Although it showed no degradation after 15 minutes of gaseous OF_2 exposure, a second sample, exposed for 30 minutes, became tacky.

3.2. Twenty-Four Hour Gaseous OF_2 Tests

Tensile specimens were prepared from the materials which survived the preliminary tests and were exposed to OF_2 gas at 50 psig for twenty-four hours. The specimens were weighed and measured before and after exposure. The specimens were weighed after removal from the bomb, and then placed in a vacuum oven at 75°C for twenty-two hours. When temperature equilibrium was regained, specimens were again weighed. Durometer readings were also taken before and after exposure. Since the twenty-four hour gas phase exposure was a preliminary test to further eliminate questionable materials, single specimens were used.

3.2.1. Experimental Procedure

The cleaned and weighed specimens were hung on a rack inside the stainless steel bomb. The bomb was approximately 4" I.D. by 6" deep and the bolted cover was sealed with a teflon gasket (Figure 4). The assembled bomb was then connected to a manifold located behind a steel barricade. The bomb and lines were evacuated before being filled with gaseous OF_2 to the desired pressure of 50 psig. All valves were controlled from the outside of the barricade and the bomb pressure was visually monitored by a gauge located behind a window in the barricade wall. The set-up is shown schematically in Figure 5. After twenty-four hours, the OF_2 was vented off and the entire system flushed with nitrogen. The bomb was then removed, opened, and the specimens inspected.

3.2.2. Results

The results obtained from this series of tests are listed in Table 9. In the third run, a slight pressure increase was noted after the first hour, apparently indicative of some reaction. This was confirmed on inspection after the run was completed. The gauge was found to be out of calibration which indicated that a pressure had been reached in excess of 100 psig, the upper limit of the compound gauge. Upon venting the bomb after Run #3, heavy smoke was noted in the vent exit. Examination of the specimens indicated that the silicone elastomer, K-1920, from Union Carbide, apparently ignited. The remaining samples, while damaged, were not destroyed. Fresh specimens of these damaged materials were therefore re-tested in Run #4. On the basis of these twenty-four hour gaseous OF_2 tests, nine additional materials were eliminated from further consideration.

3.3. Seven Day Gaseous OF_2 Tests - Procedure

Fresh specimens of the materials that satisfactorily passed the one day OF_2 exposure tests were exposed for seven days. The same equipment and techniques were used as in the one day test except that all materials were represented by duplicate tensile specimens. The initial bomb pressure was 50 psig. A minimum of two pressure readings were taken daily during this period. If the pressure dropped to less than 45 psig, the bomb was re-pressurized to 50 psig by adding more OF_2 .

Pressure drops were attributed to either possible OF_2 absorption by the specimens or minute leaks in the system.

As in the twenty-four hour tests all specimens were weighed and measured before and after exposure. In addition, hardness measurements were taken with a Shore Durometer. Tensile tests were conducted with these exposed specimens and compared to the tensile strength of the unexposed material. The complete tensile testing program will be covered in a separate section (3.5) of this report.

3.3.1. Results

The results of the seven day exposure to OF_2 vapor are shown in Table 10. Fourteen materials were thus tested and all appeared to be unaffected by the OF_2 and were therefore included in the liquid OF_2 compatibility phase of this program.

3.4. Liquid OF_2 Storage Tests

Materials which had satisfactorily passed the gas phase OF_2 storage tests were subjected to liquid OF_2 storage. Two series of tests were conducted, 48 hours and seven days exposure, respectively. In no test was a specimen attacked or affected by the OF_2 . The majority of specimens showed negligible weight changes and no change in appearance or hardness.

3.4.1.

48 Hour Tests - Equipment and Procedure

The cleaned and weighed specimens were hung on a rack inside the stainless steel bomb. The bombs were the same as were used in the gaseous OF_2 tests. Since the handling of large quantities of liquid OF_2 was hazardous, the work was performed inside a high pressure cubicle. All filling and venting operations were performed by remotely controlled valving. A schematic of the system is shown in Figure 6.

The bomb was attached to the manifold and the entire system evacuated, sealed off, and passivated for 24 hours with OF_2 gas at 10 psig. Since the system in normal operation would not be exposed to OF_2 at higher pressures, it was felt that this passivation treatment would be adequate. Referring to Figure 6, Valves 1 to 5 were remotely operated through the cubicle walls. Valves 3, 4, and 5, which were in the most critical locations, were Nupro "BG" series, all stainless steel with welded bellows. These valves were leak-tight both under high vacuum and at pressures to 1000 psig. Valves 1 and 2, Monel Whitey Valves No. 1KS4, were suitable for their use in nitrogen flow control. Some difficulty from galling was encountered since the valves had been degreased before they were placed in service. The Whitey valves required frequent replacement and were eventually replaced with Hoke Y 343 needle valves which performed satisfactorily for the remaining tests.

Valve 6 was a globe valve which controlled the house nitrogen from outside the cubicle. Valves 7 and 8 were Hoke M 343's on which the pipe threaded connections had been back brazed to prevent leaks. These valves, which controlled the OF_2 , were located in another cubicle along with the OF_2 supply cylinder.

The evacuated bomb, after being submerged in the LN_2 Dewar, was charged with OF_2 . The quantity of OF_2 transferred was measured by the pressure differential at the OF_2 cylinder gauge. For the forty-eight hour tests the specimens were half immersed in the OF_2 . This required approximately 1-1/2 lbs. of OF_2 per run.

The LN_2 level in the Dewar was maintained by means of a level control which regulated the flow from a LN_2 reservoir located outside the cubicle. Tests showed that the immersed bomb could be kept at LN_2 temperature for over 90 hours using a 50-liter LN_2 reservoir. Another interesting feature was the use of a compound gauge equipped with electric contacts. A pressure rise in the bomb arising from loss of external coolant or an internal chemical reaction would ring an alarm when the pre-set pressure was reached. As an added precaution, a burst disc set for sixty pounds was tied into the system. Should this disc burst, the gases in the bomb would then be vented to a charcoal burner where the OF_2 would be decomposed. All vent gases were likewise normally directed through this burner when the bomb and lines were vented or purged.

When a test had been completed, the LN_2 flow was cut off, the Dewar was lowered by an electrically operated jack, and the bomb was allowed to warm. The vent valve (No. 4) was not opened until the bomb pressure exceeded atmospheric to prevent entry of air into the bomb. In addition, a small trickle of nitrogen (Valve #1) was sent through the vent line before venting the bomb to remove any gases or moisture that might react with the OF_2 .

Wood charcoal of small uniform size was used in the burner. Smooth and prompt initial ignition was assured by adding a little grease to the charcoal near the OF_2 inlet. After the initial ignition several re-starts were made and in every case the ignition was smooth and quiet. The charcoal burner performed well for the first two runs, but the inlet tube burned out during the venting of OF_2 from the third run. This inlet, a 1-inch Monel pipe, burned back to the outside of the burner and vaporized both firebrick and the burner wall adjacent to the pipe. The inlet was therefore replaced by a water-cooled, jacketed copper inlet (Figure 7) which worked very well for the remainder of the storage tests.

3.4.2. Results of 48 Hour Tests

The results of these 48-hour tests in liquid OF_2 are shown in Table 11. None of the specimens appeared to be affected by the OF_2 , and no differences could be seen between the submerged and unsubmerged sections of the specimens

On the basis of weight changes, Halon TFE G-50, Halon TFE G-80, (normal, high and low crystallinity), Teflon 7, TFE, CTFE, FEP and Aclar specimens were the least affected by OF_2 . Teflon 5, Viton 985 and RM 618 appeared to be somewhat less resistant. These materials had been more completely identified previously in Table 7.

3.4.3. Seven Day Liquid OF_2 Tests - Equipment and Procedure

Upon completion of the 48-hour tests, the seven day exposure tests were initiated using the same equipment but with one important difference in procedure. The bomb was charged with 3 lbs. of OF_2 for each 7-day run and the specimens were therefore totally immersed. In the shorter (48 hour) exposure tests, 1-1/2 lbs. of OF_2 were used per run and the specimens were half immersed.

In the seven day test, generally four specimens of each of 14 materials were exposed. Although a 50-liter Dewar of LN_2 was normally more than sufficient to supply the set-up over a weekend, two runs were aborted and had to be repeated because of LN_2 feed problems.

3.4.4. Results

A total of 57 specimens involving 14 materials were exposed for a minimum of seven days. In no case was there any change in appearance or hardness. The complete list of materials with weight changes is shown in Table 12.

The first run contained the high- and low-crystallinity Halon TFE G-80 specimens. These particular specimens were exposed for approximately 102 hours when the previously mentioned run failure occurred. After reweighing they were replaced in the bomb with fresh OF_2 and exposed for seven additional days, making a total of approximately eleven days. The second run contained Halon TFE G-50 and G-80, and Teflon 5 and 7. These four materials were also exposed for 4 days followed by an additional seven days as in the first run. However, they were not reweighed after the four day exposure. On the basis of weight change, the Viton 985 and RM 618 showed that they were affected by the OF_2 somewhat more than the other materials. However, one FEP specimen and two CTFE specimens also showed some moderate weight changes. Since other specimens of these materials showed negligible changes in weight, the possibility of weighing error was considered. However, a review of the weighing and handling technique appeared to preclude this explanation.

3.4.5. Liquid OF_2 Storage Tests - Conclusion

Based on weight change, hardness, and appearance, no material exposed to liquid OF_2 could be considered to be completely incompatible with OF_2 under static conditions. However, some materials did appear to have slightly better resistance than others. Durometer readings taken on all specimens before and after exposure to liquid OF_2 showed no changes. These measurements, using either a Shore A

or D durometer, were made in accordance with ASTM Spec. D 1706-61. We believe that the seven day exposure as conducted was sufficient to provide significant results. We do not feel that exposures for longer periods (months or years) would have sufficiently greater significance to justify the increased cost and time involved in such tests. The series of liquid storage tests took somewhat longer to complete than originally programmed owing to the two runs which were aborted and repeated, the need to replace valves, and the burnout and repair of the charcoal burner. However, the system as designed and modified is extremely efficient and safe, and with little or no further modification can be used for liquid OF_2 storage tests of any duration at various temperatures and pressures.

3.5. Tensile Tests

Tensile tests were performed both at cryogenic and ambient temperatures, using OF_2 -exposed and unexposed specimens. Materials referred to in this section are more completely identified in Table 7.

3.5.1. Tensile Testing at Ambient Temperature - Procedure

All specimens tested were fabricated to ASTM die "C" tensile specifications and pulled with an Instron Tensile Testing machine. For this series of tests, specimens were pulled at a rate of 20"/min. when preliminary tests showed the elongation to be more than 100%. The few specimens with less than 100% elongation were pulled at a crosshead

speed of 2"/minute. For all ambient temperature tests, a chart speed of 2"/minute was used.

Certain constants were maintained for all of these tests. The room temperature was kept at $72 \pm 1^{\circ}\text{F}$ with a relative humidity of $50 \pm 1\%$. The initial jaw gap was 2" for all specimens and the elongation was calculated on the basis of the final jaw gap when the specimen broke.

3.5.1.1. Results

The results of these tests are shown in Table 13. Generally, the tetrafluoroethylene polymer type specimens showed no significant differences in tensile strength regardless of pretreatment. Exposure to gaseous or liquid OF_2 caused no changes in tensile strength, compared to unexposed specimens, for the following materials: TFE, FEP, Halon TFE G-80, Halon G-80(L), Teflon 5, Teflon 7, Viton 7250, Viton 985 and RM 618. The slight differences as shown in Table 13 are considered to be within the normal tensile range for the specimen. The CTFE-type materials, however, showed some significant differences. Specimens of Plaskon 2200 and 3M's CTFE showed a slight loss in strength after being exposed to liquid OF_2 for 7 days, and Halon TFE G-80(H) showed appreciable variation in tensile strength between duplicate specimens which made an accurate evaluation of its resistance to OF_2 on the basis of tensile tests impossible. Halon TFE G-50, despite negligible weight changes, showed a slightly higher strength for the specimens exposed to liquid OF_2 for 2 days. However, the 7 day liquid

OF₂-exposed specimens fell within the tensile range of both unexposed and gas-phase-exposed samples.

Elongation figures showed small differences between the several specimens of the same material. The elongation variations do not fall into any set pattern and do not appear to be a significant criterion for evaluating the effect of the OF₂ exposure. No correlations can be noted between the variations in elongation and tensile strength.

For convenience, the net weight changes of the tested specimens are also included in Table 13. Again, it can be seen from these figures that the materials generally appear unaffected.

3.5.2. Tensile Testing at Cryogenic Temperature - Procedure

The Instron Testing machine was fitted with an adapter of our own design (Figure 8) which enabled us to pull tensiles while the entire specimen and the tensile jaws were completely immersed in liquid nitrogen. These tests were all performed at -320°F. The specimens all met ASTM die "C" tensile specifications. All tensiles were pulled using a 500 lb. scale on a "D" load cell of the Instron. The crosshead speed was 2"/min. and the chart speed generally 20"/minute. A few initial tests were performed with a chart speed of 2"/min., but at this low speed the chart gave poor elongation data. Although elongation was read directly from the machine, readings were re-checked with the chart. Excellent checks were obtained between chart and machine at the 20"/min. chart speed.

3.5.2.1. Results

The results of the cryogenic tensile tests (Table 13) do not indicate that any of the TFE or CTFE-type materials were affected by OF_2 . These tests did not always show the desired degree of agreement between the exposed and the unexposed specimens, but this was probably more a reflection on the testing technique rather than an indication of material degradation.

Since the tensile tests at ambient temperature generally showed excellent correlation between exposed and control samples, the differences in tensile strength at -320° are probably not too significant.

The results of the elongation measurements likewise show no significant differences between specimens of the same material. This, together with the reported weight changes, further confirms that there was no material degradation from exposure to OF_2 .

No difficulty was found in conducting any test except for the Kynar specimens in LN_2 . At this temperature, the Kynar cracked before the jaws of the tester could be tightened sufficiently to prevent the specimen from slipping. This demonstration of extreme embrittlement alone was sufficient to eliminate Kynar from consideration for service under cryogenic conditions.

3.5.3. Crystallinity Investigation

Following completion of the tensile tests, it was noted that some duplicate samples showed appreciable variation in strength. It was also noted that there was little correlation in tensile strength between materials of similar composition. These differences were most noticeable in the tensile strength at cryogenic temperature. It was known that the crystallinity of certain materials has a very significant affect on tensile strength at -320°F . For example, Rocketdyne (Ref. 4) states that the tensile strength of Teflon of 50% crystallinity is 16,300 psi whereas 80% crystallinity material has a tensile of only 4400 psi. Likewise, Kel-F (Ref. 5) shows approximate tensile strengths of 25,000 and 15,500 psi for 40 and 70% crystallinity, respectively. Crystallinity has a negligible significance on tensile strength at ambient temperature for both classes of materials cited above.

To investigate the apparently poor correlation between the similar materials, and to compare our data with that previously published, we decided to determine the crystallinity of our TFE and CTFE-type materials. These results may be found in Table 14. The crystallinity was determined by specific gravity, density gradient and infrared spectrophotometry. It can be seen that the three methods give slightly different results. This is not unusual since each

method involves certain assumptions and different standards. Since each result reported using the density gradient technique represents the average of five separate determinations, these data are felt to be the most accurate. In the specific gravity determination only one run was made on each sample.

The sample numbers (S.N.) in Table 14 have some significance. For example, all samples with the same two numbers come from the same sheet of material. A letter A or C in the identification indicates the sample was removed from an area near an edge. The B and D denotes the sample was taken from near the center of the sheet. For example, samples 1A-3, 2A-3, 3A-5 and 4A-5 are all Halon TFE G-50, but represent four different sheets of this material from which tensile specimens were removed from near the edge of the sheet. From this information, the uniformity of crystallinity within a given sheet of plastic could be determined. However, calculations had shown that the variations indicated within a single sheet or between several sheets of the same material were not sufficient to explain the variations that were found in duplicate tensile specimens. The differences in crystallinity found between the several different tetrafluoroethylene polymers was also insufficient to account for the differences in their tensile strengths. However, the tensile strengths and crystallinity of the two monochlorotrifluoroethylene polymers correlate very well with Rocketdyne's data on Kel-F.

3.5.4.

Conclusions

Based on weight changes, tensile tests, elongation measurements, and Shore Durometer readings, specimens composed of tetrafluoroethylene (TFE), trifluorochloroethylene (CTFE) polymers, and the fluorinated ethylene-propylene copolymer (FEP) appear to be satisfactorily resistant to OF_2 under static conditions. Viton 7250, a perfluoropropylene-vinylidene fluoride copolymer, showed no loss in strength or visual evidence of degradation or attack. However, specimens exposed to gaseous OF_2 showed relatively significant weight changes indicating both absorbency and reaction with OF_2 . Tensile tests indicated severe embrittlement at -320°F since two specimens broke while being clamped into the tensile tester. Viton 985, a similar copolymer, displayed the same disqualifying characteristics as Viton 7250 but to a lesser degree. RM 618, a butaprene rubber, while showing no deterioration in strength did exhibit embrittlement and significant weight loss on final weighing. The weight loss seems to be attributed to chemical reaction with OF_2 since losses increased with the duration of exposure. Kynar as previously stated also exhibited extreme embrittlement at -320°F .

It should be noted that none of the specimens in this evaluation showed any changes in hardness (Shore Durometer type A or D) after exposure. Likewise, no significance could be attached to elongation data as evidence of degradation.

Variations in % elongations did not appear to be correlated with tensile strength or weight changes.

The testing program as performed served to establish the compatibility of certain plastic and elastomeric materials with oxygen difluoride under static conditions. The most promising materials, which included TFE, CTFE and FEP, were therefore considered for additional testing under dynamic conditions.

4. DYNAMIC TESTING OF MATERIALS IN LIQUID OF₂

It was felt that dynamic testing of the plastic materials which had previously been found to be compatible with OF₂ was necessary before these materials could be fully recommended for OF₂ service. We had therefore proposed to enlarge the scope of this contract to include a dynamic test program, the aim of which was to determine the maximum velocity at which such materials could be safely used in liquid OF₂ service.

4.1. Apparatus and Equipment

The apparatus and equipment for the OF₂ dynamic study was erected in the high pressure cubicle in which the liquid OF₂ storage tests were performed. The initiation of this program was therefore delayed until the static tests were completed. The set-up is represented schematically in Figure 9. The design reflected NASA's Plum Brook Station dynamic testing facilities and experience. However, the set-up had been adapted to our facilities and requirements. All hazardous operations were remotely controlled.

The liquid nitrogen tank was a double wall welded aluminum shell which was completely filled with a six-inch thick polyurethane foam insulation. Allied Chemical Corporation's rigid foam was chosen primarily because of its low "K" factor which is for example, approximately 1/3 that of perlite. The foam also provided additional strength and rigidity thus permitting us to select a light gauge aluminum for the tank material.

The various valves shown in the drawing (Fig. 9) have been numbered for convenience. You will note that in the following valve identification, M, A, and S stand for manually operated, air operated, and electric solenoid type valves, respectively:

<u>Valve No.</u>	<u>Type</u>
1, 2, and 4	1/4" Hoke M 343 (M)
3	1/2" Globe (M)
5, 6, 7, 10, 11	1/4" Nupro BW (M)
8, 9	1/2" Annin #1620 (A)
12, 13	1/4" Hoke (S)
14, 15	1/4" Annin (S)

Special attention was given to the design of the specimen holder shown in Figure 10. It was fabricated to maintain a tight seal regardless of whether or not the test specimen was destroyed to prevent contamination of the liquid nitrogen (LN_2) with OF_2 .

4.2. Procedure

As shown in Figure 9, the spent OF_2 was not collected but was vented directly to the charcoal burner (Figure 7) which was suitable for either fluorine or OF_2 disposal. The entire system was designed so that all exit gases, purge gases, and even the nitrogen from the LN_2 tank, could be vented through the burner. The LN_2 exit lines had been tied into the vent system to take care of the extremely remote possibility of OF_2 leaking into the LN_2 .

It should be noted that the test specimens were discs with a centered orifice of 0.0135" diameter. The system was designed to operate at pressures to 500 psi, which would achieve velocities up to 90 ft./sec. and Reynolds' Numbers in the order of 20,000 through this orifice. Initial runs were to be made at quite low pressures. A timing device had been provided to closely control the run durations which were to be for five seconds. It had been hoped that the actual flow rate could be calculated from the pressure differential in the OF₂ reservoir.

4.3. Orifice Calibration

It had been our intention to measure the flow of liquid through the orifice as a function of the pressure change in the OF₂ reservoir. It was therefore necessary to accurately measure the total volume of the cylinder and lines up to the second Annin valve. A CTFE orifice (0.0135" diam.) specimen was used for the preliminary calibration.

A known volume of water was added to the OF₂ reservoir and pressurized with nitrogen. The water was then permitted to replace the air in the lines. The water passing through the second Annin valve could then be collected and accurately measured to provide flow data for various pressures. The data collected from this calibration effort is shown in Table 15. The run data shown in this table are actually averages of two or three runs at each listed pressure increment. The difference between duplicate runs was generally less than 2% of the water flow per run, indicating very consistent results could be obtained with this set-up.

4.4. Test Procedure - Test #1

4.4.1. Cleaning

The Annin valves had been ordered in LOX clean condition and were not disassembled when received since they were to be wetted with distilled water during the calibration runs. After the calibration runs, the system was taken completely apart (except Annin valves) and all components were washed with water and detergent, rinsed with distilled water, acetone rinsed, dried with high purity N₂ and packaged in polyethylene bags until assembled. The Annin valves were given several rinses in acetone and N₂ dried.

The material used in this first test was an orifice specimen of TFE, molded by Almac, from duPont resin. A microscopic examination of the specimen had revealed some burrs at the edges of the orifice. It was therefore carefully deburred before cleaning. The specimen was washed with soap and water and rinsed thoroughly. Since the microscope revealed some specks of dirt embedded in the surface after this washing, the specimen was immersed in boiling nitric acid, followed by a water wash and rinsed in distilled water. It was next washed in acetone and then dried in a vacuum oven at 85°C for two hours. When cooled, it was weighed and the orifice measured using a microscope equipped with an appropriate reference grid.

4.4.2. Passivation

The assembled system without the test specimen was checked for leaks at 500 psi and vacuum tested overnight. When found to be tight, the system was installed in the LN_2 container in the cubicle, evacuated and then re-checked with N_2 at 500 psi. The pressure was then dropped to atmospheric and F_2 gas was slowly introduced into the system. When the exit gas was fluorine rich, the downstream Annin Valve No. 2* was closed and the pressure slowly increased to the fluorine cylinder pressure, approximately 350 psig. The system was then padded with N_2 to 450 psig and left overnight with both Annin valves closed. No pressure change was noted in the morning and the fluorine was vented to the charcoal burner. The system was completely flushed with N_2 and then removed from the cubicle to insert the specimen. When the specimen holder flanges were opened, some corrosion deposits were noted. This section was therefore removed for cleaning and the open ends of the other sections were sealed against the atmosphere. After cleaning as previously described, the re-cleaned sections were again passivated in the laboratory with F_2 at atmospheric pressure.

*The Annin valve between the OF_2 reservoir and the test specimen will be referred to as No. 1. Annin valve #2 is located downstream of the test specimen. In Figure 9 these valves are shown as Nos. 8 and 9, respectively.

4.4.3. Test Run

The test orifice was inserted and the section re-assembled. Difficulty was noted in getting the system leak tight but this was finally accomplished. The set-up was then reinstalled in the cubicle where it was re-checked at 500 psig and then evacuated overnight. The system was then filled with helium at 500 psig and LN_2 added to the trough to cover the set-up. The system was found to be tight at LN_2 temperature, after the helium pressure reached equilibrium. On the morning of the run, the system was pumped down for 3 hours with the upstream Annin opened intermittently, and the other closed. After filling the tank with sufficient LN_2 to cover the OF_2 system completely, approximately one pound of OF_2 was condensed into the liquid OF_2 reservoir, the system pressurized with helium, and the test runs started. Runs were automatically controlled by a timer and all were of 5.0 seconds duration. The initial runs were at 25 and 50 psig and then the pressure was increased in increments of approximately 50 psig for the remaining runs. The pressure for the final run was 480 psig. Pressure readings were recorded before and after the five second cycle. Gauge readability limits were about one pound. Some of the pressure drops shown could have been a result of the helium not being at equilibrium temperature at the start of the run. The nominal flows through the orifice as it was calibrated were not sufficient to account for these pressure drops. Another explanation

is that leakage may have developed across the seats of the Annin valves since some leakage was noted later when the set-up was prepared for the next test. The run data are shown in Table 16. The results shown are based on the theoretical flow through a 0.0135" orifice rather than actual measured flows.

4.4.4. Results

The test specimen was re-weighed and re-examined microscopically. It showed a loss in weight of 0.8 mg. which was considered insignificant. However, the diameter of the orifice appeared to have been reduced at the inlet edge to approximately 250 microns, while the outlet end measured about 300 microns. Initially, the orifice measured approximately 350 microns at each end. Both edges appeared discolored which was attributed to roughening by abrasion or embedded foreign matter. The surfaces which were shielded by the backup discs, remained unchanged. The specimen was washed in hot sulfuric acid to clean off any foreign particles which we had assumed to be metallic fluorides. After this treatment, the specimen appeared to be quite clean and free of visible signs of attack.

4.5. Test Procedure - Test #2

A test orifice fabricated from Halon TFE G-80 was installed in the apparatus for the next series of runs. However, the system was unable to hold pressure and a rather extensive correction procedure was initiated.

New serrations were cut on the faces of the flanges to assure better closures. Of paramount importance was our inability to correct leaks across the seats of the Annin valves. With the aid of a technical representative of Annin, we completely disassembled the valves and found corrosion on the several seats, stems and bellows of the valves. A detailed drawing of these valves has been reproduced in Figure 11. The various valve components have been identified by numbers as shown in Table 17. The valve corrosion was attributed to our failure to remove all traces of moisture from the valves after the calibration runs. Replacement parts were ordered but because of poor delivery, the stems were re-machined in our shop to Annin specifications.

Meanwhile, all the parts of the system except the OF_2 reservoir, which remained sealed to the atmosphere, had been re-cleaned. The pieces were sonic washed in a detergent solution for several hours, rinsed repeatedly in distilled water, acetone rinsed, and dried at 150°C in a vacuum oven. The replacement seats and gaskets were washed with detergent, distilled water rinsed, acetone rinsed, air dried, and bagged after cleaning until needed. The replacement bellows which we received was ordered LOX clean, but it had been unbagged and packed in excelsior and shredded paper. The open end was not taped over to prevent shreds of packing material from entering the bellows. The interior of the

assembled bellows could not be examined since there was only a slight clearance between the welded shaft and the bellows. The bellows were filled repeatedly with clean acetone to rinse out any extraneous material that might have entered. No contamination was disclosed by this cleaning and it was therefore assumed to be clean. The bellows was then drained and dried in a vacuum oven at 150°C for three hours. When cool, the bellows was flushed with fluorine in the laboratory as were all the other valve parts which had been removed from the vacuum oven. All parts after fluorine treatment were flushed with N₂ and immediately bagged until assembled.

The valves had been reassembled and installed in the system with the new bellows in Annin #1. The best looking of the two used bellows was placed in Annin #2. Pressure tests indicated there was still leaking across the seats of the Annin valves. Annin #2 was made tight by stem adjustments but this procedure failed to completely stop the leak in valve #1. Annin's representative again came to help us and found a very fine scratch on the stem and seat. The next morning the stem and seat were re-machined at our shop according to Annin specifications. After cleaning, the valve was reassembled and found to be leak tight.

When the entire system was reassembled some additional minor leaks were detected using a Halogen type leak detector which was easily corrected. The system was pressurized at 480 psig and left overnight. The next day, having shown no leakage overnight, the system was installed in the cubicle. It was re-checked for pressure tightness and then evacuated, pumping continually for three hours after vacuum was reached. The system when checked early the next morning was found to have the same vacuum. However, pumping was continued and Annin No. 1 was operated intermittently while the LN_2 tank was filled. This took approximately three hours during which time the vacuum pump was operating continuously. When the proper LN_2 level was reached, the valve to the vacuum system was closed and the pump shut down. The system was then checked for tightness at LN_2 temperature. When found to be tight, OF_2 was transferred to the liquid OF_2 reservoir. It should be noted that during the entire cool down and filling procedure a helium purge was maintained through the exit lines to prevent any air or moisture from condensing in the cooled section of tubing following the second Annin valve.

During the OF_2 transfer which took about 30 minutes, Annin No. 1 was opened three times for five-minute periods to permit OF_2 to wet the system up to the second Annin.

Approximately, 0.85 lbs. of OF_2 was charged to the dynamic test system and the OF_2 was then shut off. The reservoir was slowly pressurized to 20 psig during which time Annin No. 1 was opened twice for several minutes. When the pressure was at 20 psig, the LN_2 level was checked and sufficient LN_2 added to completely cover the Annin body flange which secured the valve extension body.

The cubicle was then sealed, all valves checked for proper position and the pressure raised to 30 psig with Annin No. 1 open. Annin No. 1 was then closed, and both Annin valves were set for timer operation. The timer button was pressed which opened both valves simultaneously for five seconds. The operator was at the same time closely watching the pressure gauge on the system through a peep-hole through the cubicle wall. The button was still being pressed when a severe explosion occurred.

4.6. Explosion Report

4.6.1. Summary

On Friday, December 18, 1964 at 12:10P.M., a severe explosion occurred when the first dynamic run of Test No. 2 was initiated. Parts of the test equipment were severely damaged and the liquid nitrogen bath was completely destroyed. However, nobody in the area sustained the slightest injury. The investigator who initiated the test was looking into the cubicle as the explosion occurred. Some flashes were observed but no movement of the pressure gauge pointer was noted. The blast as felt through the peep-hole was

severe but its impact was absorbed by the safety glasses which were worn. The building was quickly evacuated of all personnel as some white fumes (N_2) started to seep around the cubicle door and through the peep-hole. Personnel working in the vicinity reported the explosion was a loud, sharp report. However, nobody detected any OF_2 fumes up to the time they had left the building.

The building was re-entered by personnel wearing air packs to make sure everyone had left and also to start exhaust fans in all the cubicles. The building was entered periodically and the cubicle checked to make sure no OF_2 was trapped in the reservoir. Finally, at 4:00P.M., the elbow leading from the OF_2 reservoir was seen to be split thus eliminating any possible secondary explosion from pressure buildup. The building was then secured for the weekend against any visitors and allowed to air out. Monday morning there was absolutely no traces of odor in the test cubicle or elsewhere in the building and work was resumed.

4.6.2. Damage Evaluation

The damage can best be described with the aid of pictures which were taken after the incident. These pictures may be found in the Appendix of this report. The blast panel of the cubicle had blown out (Exhibit A) and the juxtapositioned panels of the adjacent cubicles were loosened by the force of the explosion.

Inside the cubicle the aluminum LN₂ tank had been peeled open and wedged against both walls. (Exhibit B). Much of the polyurethane insulation had been pulverized and covered everything with a layer of fine dust. The test set-up seemed virtually intact although the elbow burnout was visible and burn marks were noted on the No. 1 Annin valve (Exhibits C & D). The wiring to this valve had been burned through but the second Annin valve was still in operating condition. None of the quarter inch copper or stainless steel feed lines were damaged. (Exhibit E). In fact, the glass cover of the pressure gauge was not even cracked. The exit line, however, was broken where it had been reduced to 1/4" to accommodate a 1/4" Nupro valve. The break resulted from the twisting of the set-up when the frame mountings were blown loose. The Annin valves had been attached to the frame with "C" clamps, one of which can be seen in Exhibit E.

The stainless steel dynamic set-up was removed from the cubicle for examination. Exhibit F shows the complete system as removed from the cubicle after the burnout except that the loose insulation and dust had been removed. Exhibits G & H, respectively, show Annin valve No. 1 which was severely damaged by the burnout, and Annin No. 2 which was undamaged. It should be noted therefore that all burned valve components referred to in this report were taken from Annin No. 1. (Valve 8 as shown in Figure 9).

The system was dismantled and the damaged components were photographed. The 1/2" stainless steel tubing leading from the OF₂ reservoir was burned out where it had been bent. This bend, which is also referred to as an elbow in this report, is shown in Exhibits I & J. The opposite edges of the burnout show a remarkable degree of symmetry. A closeup of the face of the flange downstream of this elbow is shown in Exhibit K. You can see that half of this face is severely burned while the other half still shows bright serrations. A small piece of the aluminum gasket was found on the unburned face section.

The burned Annin valve was completely dismantled and various components photographed. These valve components have been further identified by part number as shown on Figure 11. Exhibit L shows the severely burned flange (part 10) face at the inlet side of the valve. This flange which was mated to the flange shown in Exhibit K was more severely damaged. Exhibit M shows the opening in the valve body section through which the valve stem rode. This section and the flange (part 25) were severely attacked. The mating flange (part 23) was also damaged as shown in Exhibit N. The body extension (part 15) is shown in Exhibit O. The bottom end and the interior show moderate damage. The threads of the plug and the tapped hole were also burned.

Downstream of the valve, the specimen holder flange faces were damaged. (Exhibit P). The outlet side, which has the small opening, is shown on the left. Figure 10 is a detailed drawing of the specimen holder with the OF_2 flow direction, as installed, from left to right. Exhibit Q shows the mating flange to the Annin valve #1 exit on the right as compared to the unaffected flange which was mated to the inlet of Annin valve No. 2. This picture shows that no damage occurred downstream of the specimen holder. It should be noted the two flanges shown in the background of Exhibit Q are the flanges seen in Exhibit P and vice versa.

All of the bolts fastening the various burned flanges showed some attack. The bolts securing the valve body extension flanges were deeply grooved. (Exhibit R).

The valve stem plug and seat are shown in Exhibit S. The stem had burned off the bellows assembly. In fact, the lock nut (Figure 11, part 11) was completely destroyed. The copper seat as pictured shows the face which mated to the plug. The opposite face of the valve seat showed no deposits or corrosion.

The most severely damaged section of the valve was probably the bellows assembly (Figures T and U). As can be seen, the corrugated bellows was almost completely destroyed. The two gaskets which sealed the bellows to the valve body (parts 13 and 14) were also destroyed as were aluminum gaskets between the other flange faces.

The inlet side of test specimen is shown in Exhibit V. Except for the orifice enlargement and a very slight chamfer at the edge of the orifice it remained relatively unaffected. The specimen weight loss was 0.1265 grams. The thrust ring which faced the specimen is shown in Exhibit W. This piece is shown in Figure 10 as a stainless steel washer. The picture shows that appreciable erosion occurred on the face of this part. The reverse side which was the upstream side surprisingly showed no erosion.

In order to assess the amount of metal consumed in this burnout, the weights of various components were compared to identical unaffected parts. These weight losses together with several measurements have been listed in Table 18. The losses are of course conservative since weighings were made with the considerable metallic fluoride deposits still coating the components.

4.6.3. Analytical Data

Some of the residual corrosion by-products were analyzed. X-ray analysis of the whitish deposit on the bellows shaft showed FeF_3 and NiF_2 . Spectrographic analysis of this material, as well as the deposit removed from the base of the bellows, showed Fe and Ni as major constituents, and Cu and Cr as minor constituents. No oxides were found in this material. Downstream of the damaged Annin, some black deposit removed from the face of the specimen backup disc was found to be iron oxide.

4.6.4.

Calculations

Based on the determined amounts of material consumed and the assumption that the lost material had reacted primarily to form fluorides, the energy released in this burn-out had been calculated. It should be noted that significant quantities of stainless steel were consumed at the several flange faces and at the elbow. These weight losses could not be measured and are therefore not included in these calculations.

By weighing the burned metal parts and comparing the weights with new parts, it was determined that approximately 180 grams of stainless steel, copper and aluminum reacted with the OF_2 . By stoichiometry this accounts for approximately 67% of the total OF_2 originally charged into the system. No oxides were found by chemical analysis in the residues taken from the burned components. Therefore, in writing the chemical reactions it was assumed all the metals were converted to fluorides. With this as a basis, a thermodynamic analysis indicated that approximately 767 kilo-calories of heat were evolved in the process. Taking into consideration the sensible heats, and the fusion and vaporization processes, the maximum temperature achieved is estimated to be approximately 3800°C .

A stress analysis on the bent shaft which connected the bellows to the air motor of the Annin valve indicated that a minimum loading of about 3000 pounds was required to cause the failure of this part by bending.

Since the pressure area on this shaft is something less than 1 square inch, pressures in excess of 3600 psi must have been achieved in the valve area of the system.

4.6.5. Conclusions

It is felt that this occurrence is another example of a so called explosive burnout phenomena. The evidence indicates that the plastic specimen did not trigger the burnout. The orifice enlargement from 0.0135" I.D. to approximately 1/8" was a result of the high temperatures and pressures generated within the system. The point of initiation is suspected to be within the bellows. The fact that it is the most totally destroyed component of the valve would tend to substantiate this conclusion. However, consideration must be given to the fact that its physical configuration would tend to cause it to react more vigorously than say the relatively bulky valve body. The lower section of the bellows and the stem assembly show evidence of vigorous attack. The stem was burned from the bellows assembly and the lock-nut was totally consumed.

The vigor of the reaction indicates that it occurred virtually spontaneously. The resulting high temperatures and pressures caused burnout at the flanges and the tubing elbow. The release of the vaporized fluorides, molten metal and other hot gaseous OF_2 by-products instantaneously vaporized sufficient liquid nitrogen to cause tremendous

hydrostatic pressure which exploded the LN_2 tank. There was no evidence of burning of either the insulation or aluminum except for some finely divided particles of polyurethane that was found on the upper walls of the cubicle. The explosion of the aluminum tank is believed to be largely responsible for the loud noise.

The cause of this burnout cannot be given with complete certainty. It had been noted that this run was not preceded by the fluorine passivation of the assembled set-up. The individual components however had been passivated with fluorine. Care was taken to protect the cleanliness of all components from the time of passivation through the assembly of the various units. The mechanic who reassembled the valves was advised of the need for extreme cleanliness and it is assumed he performed his task according to the necessary standards. The background of the bellows causes it to be suspect. The poor packaging procedure used by Annin in shipping this bellows would normally have caused us to reject it for use in this service. The fact that this phase of the program had been beset with innumerable delays due to leakage and poor delivery of replacement parts persuaded us to use this bellows. The use of this bellows combined with the fact that passivation was not performed at working pressures were calculated risks taken to save time since passivation of the completely assembled system required two days. The system must be passivated without the test specimen. After passivation the set-up must be removed

from the cubicle to insert the specimen and again pressure tested. When tight it was again mounted in the cubicle and again pressure tested. This laborious technique was necessary since the force applied to the flange bolts when the set-up was in the LN_2 trough was insufficient to stop the leaks. Whether a different bellows or full passivation would have prevented this incident is of course problematical.

As a result of this explosion, the remaining work on the dynamic program was postponed and our efforts were directed toward an investigation of the so called OF_2 explosive burnout phenomena which is described in Section 5 of this report.

It should be noted that the required dynamic exposures of plastic orifice specimens to liquid OF_2 were completed as part of a second OF_2 Research Study. This work is reported in complete detail in the Final Report, Contract No. NAS 3-6298, "Oxygen Difluoride Research Study".* In this program various TFE, CTFE and FEP materials were exposed to liquid OF_2 at pressures up to 500 psig. All materials tested appeared to be compatible with liquid OF_2 under the test conditions.

*NASA CR 72357

5. INVESTIGATION OF EXPLOSIVE BURNOUT

Incidents have been reported in which OF_2 has suddenly and unexpectedly reacted quite vigorously. Such reactions have often resulted in burnouts of the valving, lines and other sundry hardware at several points in the system. The rapidity with which this phenomenon has occurred has caused it to be called, "explosive burnout." We have therefore conducted an investigation to determine whether burnout can be initiated in a properly designed, cleaned and passivated vessel by the sudden release of energy into OF_2 at elevated pressures. In this investigation the energy was provided by Pyrofuse wires which were ignited inside the OF_2 filled reactor.

5.1. Apparatus and Equipment

The apparatus for this investigation was installed in a high pressure cubicle. A schematic representation of the set-up is shown in Figure 12. The manifold consisted of six Pressure Products, Monel 30,000 psi needle valves which were used in conjunction with Pressure Products, cone type, high pressure service lines and fittings. The remaining valves, each indicated in the drawing by a circled X, were Hoke 343M needle valves to which were attached Swagelok fittings and 1/4" copper lines.

The reactor or test bomb was a modified double opening Hoke, Monel, 150 cc. cylinder rated at 5000 psi.

Modifications consisted of welding fittings on opposite sides of the bomb. One fitting was used to attach the electrode adapter while the other provided a connection to a Monel burst disc adapter. A 2000 psi burst disc was selected since the upper limit for these tests was 1500 psi. After the fittings had been welded, the vessel was hydrostatically tested at 2000 psi and found to be satisfactory. A picture of the modified bomb is shown in Figure 13.

The OF_2 condenser was a similar Hoke cylinder except that it had a single opening. The containers used to sample the residual gases after the Pyrofuse wire ignitions were 500 cc. stainless steel cylinders. The large volume of these receivers permitted us to obtain adequate samples of gas at reduced pressures. Monel components were selected for the sections of the system which were exposed to OF_2 at elevated pressures. To mitigate the possible effects of contaminants in the system, the use of pipe dope or Teflon pipe tape was avoided. Therefore all threaded connections in the set-up were brazed or welded to assure leak tight closures.

To prevent atmospheric contamination with the toxic OF_2 , all exit and purge lines were vented into a charcoal burner. This method of disposal had previously been demonstrated to be very effective in decontaminating OF_2 .

The Pyrofuze wire and the electrode adapters used in this study are described in detail in the succeeding sections of this report.

5.1.1. Pyrofuze Wire

The energy source selected for this investigation was Pyrofuze wire, manufactured by Pyrofuze Corporation, Mt. Vernon, New York. Pyrofuze wire is a bimetallic of palladium and aluminum, the former constitutes an outer shell and the latter an inner core. When the elements are heated to the reaction temperature, the metals alloy vigorously and exothermically. The reaction temperature is 2800°C and one gram of Pyrofuze evolves 325 calories upon alloying. A preliminary investigation indicated a one-inch length of 10 mil diameter wire would be the most suitable size for the first series of tests. This length of wire weighed approximately 9.5 mg. and its ignition would release approximately 3.1 calories to the OF_2 in approximately 1-2 milliseconds.

The Pyrofuze had been tested for compatibility with both fluorine and OF_2 since it would be exposed to these gases during the testing program. A sample exposed to a gaseous flow of OF_2 for two hours showed no sign of attack by this oxidizer. The wire was then placed in a flow of gaseous fluorine for two hours. At the end of this period, it showed a very light golden color and a weight gain of 0.3 mg. The next morning, the color was gone and the wire had lost 0.6 mg. The wire was re-exposed to F_2 for

four more hours, but this time, there was no change in weight or appearance. Ignition tests on this treated wire showed it to ignite as readily and completely as unexposed wire.

The Pyrofuze was available as both wire and foil. In the second series of tests which involved larger releases of energy, foil was used to meet these requirements. The composition and energy release per gram are identical for both the wire and foil.

5.1.2. Electrode Adapter

An electrode adapter was manufactured to our design by Electrical Industries, Murray Hill, New Jersey. A picture of this electrode with the Pyrofuze wire and foil attached is shown in Figure 14. The Monel body was fabricated by our shop and sent to Electric Industries where the electrodes were hermetically sealed into the provided blank.

The sealing material was a glass, reportedly similar to Pyrex, but whose exact composition was not revealed. The seals generally tended to leak after two or three runs. We therefore consumed approximately a dozen adapters during the course of this program.

The Monel adapters were machined to mate with the recessed or seat half of a 1/2" pipe union which had been welded to the bomb (Figure 13). A leak tight metal to metal seal was thus effected.

It should be noted that at the concept of this study consideration had been given to using a spark plug as the energizer but the spark per se was not considered to be a very accurate or reproducible energy source. We therefore decided to use the spark plug electrodes as terminals for our Pyrofuze wire. To our surprise we found that the spark plug seal leaked at high pressure. We discussed this problem with Champion Spark Plug Co., Toledo, Ohio who confirmed that spark plugs are not hermetically sealed. Since no satisfactory units were commercially available, we were therefore obliged to design an adapter that would meet the requirements of this investigation.

5.1.3. Cleaning Procedure

In view of the burnout that occurred during the previously described dynamic study, punctilious attention was given to both the cleaning and passivation of the apparatus for this investigation. All lines, valves, fittings and cylinders used in this set-up had been subjected to a very thorough cleaning and inspection before installation. The following cleaning procedure was used for the initial preparation of the several components:

- (1) Cleaning for 5 minutes in detergent (Joy) solution of hot water.
- (2) Rinsed with hot water until free of detergent.
- (3) Rinsed with distilled water.
- (4) Rinsed with methanol to remove water.
- (5) Final rinse 5 minutes in Genosolv D.
- (6) Dried 2 hours at 75°C in vacuum oven.

Note that steps (1) and (5) were performed with ultrasonic agitation. The above procedure was used for the small items such as tubing, valves and fittings. The several cylinders or bombs were cleaned in the same manner except cleaning (1) was performed for 1/2 hour and the drying (6) was continued overnight. To avoid recleaning, the system was sealed off between runs. After the initial assembly the only lines opened were those connecting the reactor and the sample receiver. When these units were removed, the lines were immediately sealed with caps or plugs to prevent contamination. The only unit actually handled was the adapter when the Pyrofuze wire was attached. The adapter was then washed with acetone and rinsed in Genesolve D followed by vacuum oven drying before replacement in the reactor.

5.1.4. Passivation Procedure

When the assembled system was found to be completely leak tight, it was purged with high purity nitrogen and then evacuated overnight. The evacuated system was then filled to atmospheric pressure with H.P. nitrogen and followed with a slow purge of fluorine for 15 minutes. The vent valves were then closed and the system was charged with fluorine to 150 psi. After two minutes, the fluorine was padded with nitrogen to 400 psi and allowed to remain at this pressure overnight. Since it then had been found necessary to relocate a gauge to improve

readability, the system was repassivated after this change was made. This time the system was charged with fluorine at 150 psig and allowed to remain at this pressure over a weekend.

Although every precaution had been taken to prevent air or moisture from entering the system during down time, the entire system was again passivated before each and every run. This passivation procedure consisted of flushing the system with fluorine gas for several minutes followed by pressurization with fluorine to the actual working pressure of the specific run. Since many runs were made above the F_2 cylinder pressure, sufficient fluorine was condensed in the system condenser and allowed to vaporize to obtain the desired pressure. This pressure was then held for a minimum of approximately 1/2 hour. After passivation, the fluorine was vented to the charcoal burner and the system purged with nitrogen before evacuating. The system was then kept evacuated with the vacuum pump on until it was to be charged with OF_2 . It should be noted that prior to each passivation, the entire system was pressure tested for leaks with nitrogen in excess of the scheduled working pressure.

5.2.

Test Procedure

Two series of runs were made during this investigation. The first series involved the ignition of a one-inch length of 10 mil Pyrofuze wire in gaseous OF_2 at several pressure increments ranging from 60 to 1500 psi. This length of wire released 3.1 calories upon ignition. For the second series a combination of wire and foil was used to obtain a higher calorie output on ignition. The wire and foil were accurately weighed and the calorie release calculated. One gram of Pyrofuze yields 325 calories on ignition.

The electrodes of the adapter were attached to an AC power source with the voltage output controlled at 10 volts by a powerstat. This voltage had been found more than adequate to obtain instantaneous Pyrofuze ignition. The circuit continuity was checked with a Simpson Ohm Meter both before and after the wire was ignited to make sure it had been energized.

When low pressure runs were made the gaseous OF_2 was introduced to the evacuated system directly from the OF_2 supply cylinder to the reactor. To obtain pressures greater than OF_2 cylinder pressure, the required amount of OF_2 was charged to the condenser as shown in Figure 12.

The remotely controlled electrically operated jack was then lowered and the OF_2 vaporized until the required pressure was reached in the reactor. When this was attained, the reactor was sealed off and the residual OF_2 in the condenser was either returned to the main cylinder or vented to the charcoal burner.

For all runs the initial and final pressures have been reported and any unusual activity noted. After the runs were completed, samples of the residual gas in the reactor were analyzed by various methods. The composition of this gas and the per cent of OF_2 decomposition were then calculated on the basis of the analytical data as well as on the basis of pressure differential for the run. Oxygen difluoride decomposes to fluorine and oxygen



yielding 1-1/2 moles of gases per mole of OF_2 . Therefore OF_2 decomposition was indicated by an increase in pressure as well as the presence of oxygen in the residual gas. One method of analysis was an oxygen determination by mass spectroscopy. It was felt that the oxygen analysis would be a better criterion than fluorine content for the determination of the OF_2 decomposition since the fluorine was more apt to react with the Pyrofuze on ignition.

In addition despite the careful passivation procedure, there was a possibility of a reaction between the fluorine and various components of the system as well as with the analytical equipment. This indicated that the fluorine content of the gas would not be an accurate indication of OF_2 decomposition.

5.2.1. Series 1 - Preliminary Tests

In this series of tests, the energy source, approximately 9.5 mg. of Pyrofuze, was consistent for each run. This amount of wire released 3.1 calories to the OF_2 on ignition. Each successive run was conducted at a higher pressure increment.

5.2.2. Series 2 - Final Tests

In this series of tests which were conducted at 300 and 600 psig, the energy input to the OF_2 was considerably greater than the preliminary tests ranging from 35.5 to 264.6 calories. This last amount released in OF_2 at 600 psig caused a peak pressure at ignition of almost 1800 psig, approaching the upper pressure limit of our set-up.

5.3. Experimental Data

A total of 16 runs were made. For convenience, the runs have been numbered consecutively. Runs 1 thru 11 however are considered the Series 1 or preliminary tests as described in Section 5.2.1. The remaining runs are the Series 2 (Section 5.2.2) tests. All data have been summarized and reported in Tables 19 and 20.

Run #1.——In this initial run the reactor was charged with OF_2 at 60 psig. No pressure change was observed when the Pyrofuze was ignited. Since this run was essentially made to check out the operating procedure, the residual gas was not sampled.

Run #2.——The reactor was charged with OF_2 at 200 psig. No change in pressure was noted after the Pyrofuze had ignited. Analysis of residual gas by mass spectroscopy (M.S.) indicated <1.0 mole % oxygen. Gas chromatography (G.C.) indicated 1.34% oxygen but this analysis included any nitrogen that was present.

Run #3.——Reactor was charged with OF_2 at 300 psig. When the Pyrofuze wire was ignited, the pressure gauge needle appeared to flicker. Final pressure was read as 305 psig. However M.S. analysis of gas showed no increase in oxygen content. Chromatograph analysis was not made on this sample.

Run #4.——Reactor was charged with OF_2 at 400 psig. Upon ignition, pressure increased and was read as 410 psig. However, analysis of residual gas showed no change from the previous run by mass spectroscopy. Gas chromatography was reported as 2.12% which included oxygen and nitrogen.

Run #5.——Run was made at an initial pressure of 515 psig. No change in pressure was noted after ignition. Sample showed no change in oxygen content by mass spectroscopy. Chromatograph indicated 1.78% as oxygen and nitrogen.

Run #6.——System charged with OF_2 at an initial pressure of 600 psig. No pressure change was noted when the wire was ignited. Gas sample was inadvertently lost during the analysis.

Run #7.—— OF_2 was charged to the reactor at 700 psig. After ignition the pressure was read as 701 psig. The analysis of the gas showed no significant change in oxygen content.

Run #8.——The reactor was charged with OF_2 at 800 psig. When the wire was ignited the pressure increased to 805 psig. However, analysis of the residual gas showed no significant increase in oxygen content.

Run #9.——The reactor was charged with OF_2 at 908 psig. Upon wire ignition, the needle of the pressure gauge jumped slightly but settled back at an equilibrium pressure of 910 psig. The analysis of the gas sample showed less than 1.0% oxygen.

Run #10.——Since no measurable OF_2 decomposition had been detected in the previous runs, it was decided to expedite the program by making the succeeding runs at larger pressure increments. At this time, the reactor gauge (1000 psig) was replaced with a 2000 psig gauge and the system was repassivated with fluorine at working pressure. The evacuated reactor was charged with OF_2 at 1112 psig. When the Pyrofuze wire was ignited the needle jumped but settled at 1119 psig. Analysis of the gas sample was again reported as less than 1.0% oxygen.

Run #11.—The reactor was charged with OF_2 at 1503 psig. Upon ignition the pressure increased to 1519 psig. Analysis of the gas showed approximately the same as the previous run.

Run #12.—The second series of tests wherein increasing amounts of energy were released to the OF_2 were initiated with this run. This series was intended to explore the effect of increased energy levels released while the initial pressure remained constant. For this run, 109.2 mg. of Pyrofuze wire and foil were installed in the bomb, sufficient material to release 35.5 calories on ignition. The OF_2 was charged to the reactor at 300 psig. When the foil was ignited, the pressure jumped almost instantly to approximately 375 psig but within about 15 seconds had fallen off to 320. Equilibrium was reached two minutes later at 318 psig. Analysis of the residual gas by mass spectroscopy showed a significant oxygen content, reported as 4.8%. The OF_2 content of the sample was determined to be 67.5%. These two analyses were not compatible and the gas composition was calculated based on the net pressure change in the bomb and the nature of the reactants.

When the electrode adapter was removed from the bomb, it was discovered that both electrodes had been consumed up to the hermetic seal. Preliminary tests in the laboratory had indicated that the foil could be readily ignited by wrapping it around one electrode and then connecting the foil to the other electrode with Pyrofuze wire.

The test ignition in air was accomplished with a low applied voltage and did not damage the electrodes. Apparently in the presence of OF_2 , the Pyrofuze materials ignited the electrodes. The reaction between the OF_2 and both the Pyrofuze as well as the electrodes were considered when the residual gases were evaluated.

Run #13.——In this run the energy released from the Pyrofuze foil and wire was to be double that of the previous run. Therefore, Pyrofuze weighing 199.4 mg. was used for this run. This amount of material produced 64.8 calories on ignition. The foil was attached to the electrodes by Pyrofuze wire as shown in Figure 14. It was felt that this would avoid ignition of the electrodes since only the 10 mil diameter wire was in direct contact. After evacuation the reactor was charged with OF_2 at 300 psig. Upon ignition, a peak pressure of 440 psig was observed which rapidly decayed. The final bomb pressure when temperature equilibrium was attained was 322 psig. Mass spectroscopy of the residual gas indicated 9.4% oxygen. Infrared analysis for OF_2 showed approximately 67% OF_2 . These two analyses while not in close agreement are reconcilable.

When the electrode adapter was removed from the bomb, the electrodes were found to be unaffected by the ignition. In fact, the adapter showed a weight gain of 10 mg. which could represent fluoride film deposited either during passivation or at ignition, traces of the Pd-Al alloy, or metal fluorides. The method of attaching the Pyrofuze

to the electrodes that was used in this run, was therefore used in all subsequent runs.

Run #14.—Pyrofuze foil and wire weighing 405.8 mg. were ignited in OF_2 at 300 psig. The energy released by this amount of Pyrofuze is equivalent to 132 calories. Upon ignition a peak pressure of approximately 600 psig was observed. Within one minute, the pressure had dropped to 363 psig and equilibrium was reached at 361 psig. The reactor wall temperature before ignition was 33°C . No temperature rise was noted at ignition or thereafter. A sample of the product gas was taken after equilibrium conditions had been obtained. 18.4% oxygen was determined by mass spectroscopy and 48% OF_2 in the sample by infrared analysis.

Run #15.—Pyrofuze foil and wire weighing 812.0 mg. were ignited in OF_2 at 300 psig. The energy released by this ignition was 263.9 calories. Upon ignition a peak pressure of approximately 700 psig was observed. The pressure rapidly decayed and at equilibrium, pressure was 380 psig. The external bomb wall temperature was 32° when both the initial and final pressure readings were taken.

Run #16.—Pyrofuze foil and wire weighing 814.0 mg. were ignited in OF_2 at 600 psig. The energy released was 264.6 calories. Upon ignition the pressure jumped to almost 1800 psig. It rapidly decreased and equilibrium was reached at 865 psig. Initial reactor wall temperature was 23°C and temperature at final pressure reading (865 psig) was 25°C . No significance was attached to the temperature rise since we later demonstrated that the temperature was influenced by a spotlight used to read the pressure gauge. It should be noted that despite the large peak in pressure at ignition, no noise was heard nor was any evidence of an explosion noted.

5.4.

Conclusions

These 16 tests conducted in this program indicated that OF_2 could be decomposed by thermal shock without inducing a burnout or detonation. The investigation also demonstrated that OF_2 decomposition when initiated does not necessarily proceed spontaneously to completion. On the contrary at a given pressure, decomposition is somewhat proportional to the supplied energy. The decomposition of a portion of the OF_2 does not necessarily catalyze the decomposition of the remaining OF_2 . This work also demonstrates that burnouts are not likely to occur if the proper choice of materials is made and all equipment is properly cleaned and passivated. Our set-up, which was completely fabricated from Monel, showed no signs of attack at any point despite the repeated severe service to which it was exposed.

5.4.1. Series 1

This initial series of runs in which 3.1 calories of energy were released into OF_2 at pressures ranging from 60 to 1503 psig failed to produce measurable OF_2 decomposition. Referring to Table 19, it may be seen that pressure changes were recorded for several runs. The change in pressure was assumed to be indicative of some decomposition. However, the pressure readings for Runs 1 to 6 were taken to the nearest 5 lb. increment on the gauge and are therefore not accurate. Starting with Run 7, readings were made through a small telescope which enabled us to see changes as small as 1 psig. Runs 10 and 11 show pressure increases of 7 and 16 psi respectively. Calculations for these two runs confirmed the analytical data and indicated that no significant decomposition had occurred. These calculations were based on the observed initial and final pressures in the bomb and assumed the following:

- a) Readings were taken when temperature equilibrium had been reached.
- b) No volatile gases other than OF_2 or decomposition by-products (O_2 and F_2) were present.
- c) The possible reaction between Pyrofuze and OF_2 was ignored.

With these assumptions, the pressure increase was solely due to the presence of the additional moles of gas produced by OF_2 decomposition.

In Run No. 10, the initial pressure (1112 psig) indicated .468 moles of gas in the reactor at ambient temperature (27°C). The final pressure was equivalent to .471 moles or an increase of .003 moles. Since upon decomposition, two moles of OF_2 yield one mole of oxygen and two moles of fluorine, the gas should contain .003 moles of oxygen or approximately 0.6% oxygen. This is indicative of the decomposition of 1.5% of the OF_2 .

Using the same reasoning for Run No. 11, we found an increase of .005 moles of gas which indicated approximately 0.8% oxygen in the residual gas and again indicated decomposition of approximately the same amount of OF_2 . The calculated oxygen figure checks very closely with the oxygen (<1.0%) found by mass spectroscopy.

5.4.2. Series 2

In this series of runs, larger amounts of energy were released in OF_2 at 300 and 600 psig. Since the amounts of Pyrofuze foil and wire were appreciable, the possible reaction of the OF_2 with the Pyrofuze as it ignited was considered. Pyrofuze wire and foil contains 87.38% palladium and 12.52% aluminum. Although when ignited in vacuum it forms an alloy, PdAl_3 , ignition in air caused a high percentage of metal oxide formation. It was therefore assumed that ignition in OF_2 would produce metal fluorides. All runs made in this series produced significant increases in oxygen in the product gas.

The composition of the product gas after ignition was calculated from the increased number of moles of gas present as determined from the initial and final bomb pressures. The volume of the reactor system was accurately determined to be 172cc. The following example which considers the data from Run No. 16 explains the reasoning behind this method of calculating the gas composition.

Based on $PV = nRT$, 600 psig indicated .2961 moles of gas in the bomb while at 865 psig there were .4266 moles or an increase of .1305 moles.

Since OF_2 decomposition is:



a gain of .1305 moles would indicate the decomposition of twice as many moles of OF_2 , or .2610 moles, with the formation of a like amount of F_2 . This would then give a final composition as follows:

<u>Material</u>	<u>No. Moles</u>	<u>% Vol.</u>
O_2	.1305	30.6
F_2	.2610	61.2
<u>OF_2</u>	<u>.0351</u>	<u>8.2</u>
TOTAL	.4266	100.0

This composition assumes no side reactions. Since the reaction between the ignited Pyrofuze (2800°C) and OF_2 or F_2 is possible, this has also been considered. Stoichiometrically, 814.0 mg. of Pyrofuze (87.4% Pd, 12.5% Al) could react with .0157 moles of fluorine or OF_2 . This would then give a corrected gas composition as follows:

<u>Material</u>	<u>No. Moles</u>	<u>% Vol.</u>
O_2	.1462	34.3
F_2	.2767	64.8
<u>OF_2</u>	<u>.0037</u>	<u>0.9</u>
TOTAL	.4266	100.0

The $\%\text{OF}_2$ decomposition was then calculated as follows:

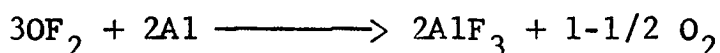
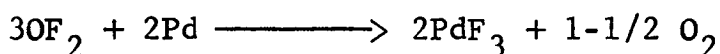
$$\frac{\text{Initial} - \text{Final Moles } \text{OF}_2}{\text{Initial Moles } \text{OF}_2} \times 100 = \%\text{OF}_2 \text{ decomposition.}$$

For Run No. 16 this calculated as 98.7% decomposition. Checking the purity of the OF_2 used for this run we found that it contained approximately 1.2% inert gases. It therefore appears the .0037 moles of OF_2 reported in the product gas was actually the inerts (O_2 , N_2 , CF_4) which of course underwent no changes under these test conditions.

The final calculated gas composition together with the % OF_2 decomposition from Runs 12 to 16 are shown in Table 20. With the exception of Run 12, the gas composition does not take into consideration any side reactions.

Run 12 was unique since the electrodes were consumed in this run. Therefore the calculated product gas composition considered the side reactions between OF_2 and the electrodes. Three separate reactions were therefore considered for this particular run since the oxygen in the product gas could have produced any or all of the following chemical reactions:

- (1) Formation of palladium and aluminum fluorides with liberation of oxygen as follows:



Since 109.2 mg. of Pyrofuze was used, the above reactions could produce .0017 moles of oxygen.

- (2) Formation of nickel and ferric fluorides when the electrodes were consumed as follows:



The weight of the electrode consumed was calculated. The initial length and diameter of the electrodes were measured from unused electrodes since all were of identical lengths.

The composition of the electrodes was determined by analysis to be 52.5% Fe and 47.5% Ni. The specific gravity was then calculated and used to estimate the weight of electrode consumed. This was found to be approximately 0.64 gms. The reaction between OF_2 and the electrodes would therefore consume .0142 moles of OF_2 producing .0071 moles of oxygen.

(3) Thermal decomposition of OF_2 as follows:



Therefore if no fluorine is consumed by side reactions, the pressure increase after ignition can only be attributed to oxygen formation. The pressure readings indicated that the total gas in the reactor increased from .1496 to .1581 moles. Assuming that neither oxides nor volatile metal fluorides were formed, the product gas must be a mixture of F_2 , O_2 and OF_2 . Considering the OF_2 consumed and the O_2 formed from reaction (1) and (2), this would indicate .0243 moles of O_2 and .1010 moles of OF_2 and the balance (.0328 moles) fluorine in the product gas.

The calculated product gas composition using these three reactions is shown in Table 20. The oxygen content (15.4%) does not check with the analytic result of 4.8%.

We therefore ran an infrared analysis of the product gas which was reported to be 67.5% OF_2 . This was considered to be in fairly good agreement with our calculated result of 63.9% OF_2 . The mass spectroscopy analysis was therefore considered to be in error and was disregarded. In fact calculations based on the reported 4.8% O_2 content yielded a final mixture in which the fluorine content was greater than twice the oxygen, a highly improbable situation.

With reference to Runs 13, 14 and 15, the product gases were also calculated. In Runs 13 and 14, the oxygen content as calculated checked quite closely with the analytical data as shown below:

	<u>Oxygen</u>	
	<u>Calculated</u>	<u>Analysis</u>
Run 13	8.9	9.4
Run 14	19.9	18.4

We have a high confidence in the reliability of our calculations and these were therefore used in preference to the analytic data which we found to be inconsistent.

5.4.3. Analytical Procedures

The oxygen analyses referred to in the preceding paragraphs mainly were those obtained by mass spectroscopy. This method as well as gas chromatography were investigated as methods for the oxygen determination in the product gases.

Although neither technique was entirely satisfactory, the mass spectrograph was considered to be the superior tool and was used for Runs 1 thru 14. The main drawback to this instrument was the fact that the lower limit of sensitivity for oxygen measurement was approximately 1%. A second but less important drawback was the inability to measure molecular fluorine with this device. On the other hand, the gas chromatograph using a silica gel column did not separate oxygen from nitrogen. Due to air leaks and the inherent inability of the instrument to pull an absolute vacuum, nitrogen was present. The mass spectrograph data were corrected for air leaks since the ratio of N_2 to O_2 from such leaks is always constant. The oxygen analysis as given had therefore been corrected according to the amount of nitrogen present. This oxygen analysis was given as mole per cent. The figures given for the gas chromatography were in area per cent. The data therefore were not readily comparable. The conversion from area to mole per cent would have necessitated preparation of standards. Since this latter method was abandoned in favor of the spectrograph, this course of action was not taken.

When we achieved significant decomposition the mass spectrograph results were initially found to be too high.

We prepared a 50:50 standard of O_2 and OF_2 . Analysis of the standard provided a correction for the analyses for Runs 12 to 14. The corrected O_2 content for Runs 13 and 14 were found to check closely with our calculated data.

Some samples were also checked by I.R. for OF_2 content. The results for Runs 12 to 14 showed a fair correlation with the calculated OF_2 . Poor agreement was found in Runs 15 and 16 but it is suspected that this was a result of not preparing a low OF_2 concentration standard with which to calibrate the infrared spectrophotometer.

As shown in Table 20, a very high pressure was noted immediately upon ignition which fell off very rapidly. This peak pressure was attributed to the heated gases in the bomb. Calculations were made for Runs 12 and 13 in which peak pressures of 375 and 440 psig were noted respectively. In neither run were the calories released by the Pyrofuze alone sufficient to explain the peak pressures noted. The calculated pressure peak in Run 12, based on the release of 35.5 calories should have been approximately 330 psig. For Run 13, with 64.8 calories, it should have peaked at 350 psig. The observed peaks were assumed to be the result of an exothermic reaction between OF_2 and the palladium-aluminum foil and/or liberated fluorine reacting with the reactor. Since the heat of formation for PdF_3 was not available, the heat release from this reaction was not calculated. However, it is

questionable whether it would be sufficient to explain the noted pressure peaks.

The phenomenon has been studied and we have concluded that the peak pressure is simply an overrun. The pressure rise being very great on ignition, the momentum of the needle carried it beyond the actual peak pressure. These peak pressures are not to be construed as indicative of the actual peak pressures in the bomb.

5.4.4. Summary

This investigation indicates that OF_2 is capable of absorbing a large release of energy without producing an explosive burnout. It should be noted however that in this investigation the energy source was not in actual contact with the wall of the container. By releasing the energy into the OF_2 or in space so to speak, the energy is apparently dissipated through the entire system and the wall did not reach ignition temperature. However, a similar release in contact with the wall could possibly have produced a burnout. In actual practice, system contamination would be on the wall of a vessel or in contact with the hardware rather than floating or suspended in the OF_2 . The electrodes were completely consumed in Run No. 12 when in contact with the Pyrofuze foil. In subsequent runs when the electrodes were not in direct contact with approximately twice as much foil, they were unharmed. This tends to indicate that burnouts are

not necessarily a result of rapid decomposition of OF_2 , but rather result from a rapid local buildup of high temperature on a surface exposed to OF_2 . Decomposition of OF_2 per se does not appear to be an explosive reaction. This work also indicates that there is a minimal energy requirement in order to initiate decomposition (Runs 1 to 11). It also indicates that the amount of decomposition is somewhat proportional to the energy release at a given pressure (Runs 12 to 15). However, where energy levels are equal, the decomposition rate is then a function of pressure.

In this program we have essentially achieved total OF_2 decomposition without causing an explosive burnout. The thermal energy to which the OF_2 has been subjected was believed to be considerably greater than could be produced by OF_2 reacting with nominal hardware contamination. It is therefore concluded that OF_2 does not necessarily decompose in an explosive manner. Burnouts can therefore be avoided if the system has been properly designed, cleaned and passivated before using for OF_2 service.

6.

REFERENCES

1. Siegmund, J. M. - "Engineering Study of Liquid Fluorine Spill Treatment Methods, Vol. I", Allied Chemical Corporation, Contract No. AF 04(611)-3366, May 4, 1959.
2. Tiner, N. A., English, W. D. and Asunmaa, S. K. "Investigation of Explosive Reactions Involving Oxygen Difluoride", Douglas Aircraft Company, AFML-TR-65-222, Contract No. AF 33(657)-9162, September 1965.
3. Siegmund, J. M., Jackson, R. B., et. al. - "Research on Materials and Methods for Decontamination of Toxic Missile Propellant Spillage", Allied Chemical Corporation, TDR No. ASD-TDR-62-64, Contract No. AF 33(616)-7672, June 1962.
4. "Final Report, Program of Testing Nonmetallic Materials at Cryogenic Temperatures", Rocketdyne, Div. of North American Aviation, Inc., Contract No. AF 04(611)-6354, December 30, 1962, pg. 71.
5. Ibid., page 80

TABLE 1
OF₂ DECONTAMINATION STUDY
GAS PHASE TESTS

<u>Material</u>	<u>%</u>	<u>OF₂ Gas</u>	<u>mg F₂</u> <u>Recovered</u>	<u>% OF₂ Decontaminated</u>
NaCl	5	21 cc	2.6	7.4
NaCl	5	21 cc	5.8	16.5
NaOH	5	21 cc	8.8	25.1
NaOH	5	21 cc	6.25	17.8
Na ₂ CO ₃	5	21 cc	2.4	6.8
Na ₂ CO ₃	5	21 cc	2.0	5.7
NaHCO ₃	5	21 cc	0.6	1.7
NaHCO ₃	5	21 cc	0.7	2.0
KOH	5	21 cc	9.6	27.4
KOH	5	21 cc	12.0	34.2
K ₂ CO ₃	5	21 cc	4.3	12.2
K ₂ CO ₃	5	21 cc	3.0	8.5
MnSO ₄	5	25 cc	0.16	0.45(b)
MnSO ₄	5	25 cc	0.14	0.40(b)
NH ₄ OH	5	25 cc	34.0	81.0 (c)
NH ₄ OH	5	25 cc	34.5	82.0 (c)
(NH ₄) ₂ CO ₃	5(a)	25 cc	7.0	20.0
(NH ₄) ₂ CO ₃	5(a)	25 cc	6.25	17.8
NaCl	5	25 cc	4.25	12.1
NaCl	5	25 cc	5.00	14.2
Urea	5	22 cc	0.60	1.7
Urea	5	22 cc	0.56	1.6
Ethanol	5	22 cc	0.35	1.0
Ethanol	5	22 cc	0.35	1.0
Methanol	5	22 cc	0.43	1.2
Methanol	5	22 cc	0.43	1.2
MgCO ₃	5	20 cc	0.38	1.08
MgCO ₃	5	20 cc	0.40	1.1
Dioxan	5	20 cc	0.38	1.08
Dioxan	5	20 cc	0.04	---
CaCl ₂	5	20 cc	2.2	6.2
CaCl ₂	5	20 cc	1.9	5.4
Na ₂ SO ₃	5	22 cc	12.0	32.4
Na ₂ SO ₃	5	22 cc	10.0	27.0
Sodium Methoxide	5	23 cc	14.0	37.9
Sodium Methoxide	5	23 cc	17.0	45.9

TABLE 1
(Continued)

<u>Material</u>	<u>%</u>	<u>OF₂ Gas</u>	<u>mg F₂ Recovered</u>	<u>% OF₂ Decontaminated</u>
Na ₂ SO ₃ in 1% NaOH	5	21 cc	19.5	52.6
Na ₂ SO ₃ in 1% NaOH	5	21 cc	17.0	45.9
KI	5	21 cc	9.4	25.4(d)
KI	5	21 cc	19.4	51.9(d)
H ₂ O	100	22 cc	0.17	0.48
H ₂ O	100	22 cc	0.15	0.40
NH ₄ OH	7-1/2	22 cc	27.0	73.0
NH ₄ OH	7-1/2	22 cc	32.0	86.0
NH ₄ OH	10	21 cc	31.0	88.5
NH ₄ OH	10	21 cc	32.0	91.0
Triethanolamine	5	20 cc	17.0	50.4
Triethanolamine	5	20 cc	21.0	61.3
Isopropylamine	5	20 cc	17.0	50.4(e)
Isopropylamine	5	20 cc	19.0	56.3(e)

- (a) 5% on NH₃ basis
- (b) Slight darkening
- (c) White fumes given off
- (d) Solution turned dark brown
- (e) Reaction accompanied by flashes and mild explosions

TABLE 2
OF 2 DECONTAMINANTS

<u>Material</u>	<u>Mfg. or Supplier</u>	<u>Grade or Ident.</u>
NaCl	(1)	B & A - Reagent Code #2226
NaOH	(1)	B & A - Reagent Code #2327
Na ₂ CO ₃	(1)	B & A - Reagent Code #2227
NaHCO ₃	(1)	B & A - Reagent Code #2202
NH ₄ OH	(1)	B & A - Reagent Code #1293
(NH ₄) ₂ CO ₃	(1)	B & A - Reagent Code #1283
CaCl ₂	(1)	B & A - Reagent Code #1502
KOH	(1)	B & A - Reagent Code #2069
K ₂ CO ₃	(1)	B & A - Reagent Code #2101
MgCO ₃	(1)	B & A - Reagent Code #1908
MnSO ₄	(1)	B & A - Reagent Code #1957
Urea	(1)	B & A - Reagent Code #2407
Ethanol	(1)	B & A - CD-19 Code #1213
Dioxan	(1)	B & A - Tech. Code #1697
Methanol	(1)	B & A - Reagent Code #1212
KI	(1)	B & A - Reagent Code #2120
Na ₂ SO ₃	(1)	B & A - Reagent Code #2301
Sodium Methoxide	(2)	Code #5943
Triethanolamine	(2)	Code #2885
Isopropylamine	(2)	Code #5470

(1) Industrial Chemicals Division, Allied Chemical Corporation.

(2) Matheson, Coleman & Bell.

TABLE 3
OF₂ DECONTAMINANTS
PRELIMINARY ECONOMIC EVALUATION

<u>Material</u>	<u>Est. Cost Per Ton</u>	<u>Approximate Effectiveness</u>	<u>Est. Cost to Neu- tralize 100 lb. OF₂</u>
NaCl	\$ 22.00	10%	\$ 550.00
NaOH	104.00	25%	1,040.00
Na ₂ CO ₃	50.00	5%	2,500.00
NaHCO ₃	50.00	2%	6,250.00
Na ₂ SO ₃	80.00	33%	600.00
Na ₂ SO ₃ in 1% NaOH	80.00 + 20.00	50%	500.00
KOH	140.00	33%	1,050.00
K ₂ CO ₃	140.00	10%	3,500.00
NH ₄ OH	45.00	80%	140.00
(NH ₄) ₂ CO ₃	760.00	20%	6,375.00
MnSO ₄	85.00	less than 2%, Economically unfeasible	
Urea	160.00	less than 2%, Economically unfeasible	
Ethanol	158.00	less than 2%, Economically unfeasible	
Methanol	88.00	less than 2%, Economically unfeasible	
Dioxan	580.00	less than 2%, Economically unfeasible	
MgCO ₃	240.00	less than 2%, Economically unfeasible	
CaCl ₂	34.00	5%	1,700.00
Sodium Methoxide	7,500.00	40%	46,875.00
KI	2,300.00	50%	11,500.00
Isopropylamine	650.00	53%	3,050.00
Triethanolamine	440.00	55%	2,000.00

NOTE: Above based on gas phase OF₂ decontamination study.

TABLE 4
OF₂ DECONTAMINATION STUDY
LIQUID PHASE TESTS

<u>Test Material</u>	<u>Concentration</u>	<u>Milliliters</u>		<u>mg OF₂ Recovered</u>	<u>% OF₂ Decontam.</u>
		<u>Sol'n.</u>	<u>OF₂</u>		
KOH	1%	200	3-1/2	5.5	.12
KOH	3%	200	3-1/2	20.0	.45
KOH	5%	200	3-1/2	19.0	.43
NH ₄ OH	1%	200	3-1/2	9.4	.21
NH ₄ OH	3%	200	3-1/2	30.0	.69
NH ₄ OH	5%	200	3-1/2	44.0	.99
NH ₄ OH	5%	200	5	21.0	.46
NH ₄ OH	5%	100	5	19.0	.43
Na ₂ SO ₃ (a)	5%	200	3-1/2	130.	2.9
H ₂ O	100%	200	3-1/2	3.4	.07
(NH ₄) ₂ CO ₃	3% (b)	200	3-1/2	3.9	.08
Methanol	5%	200	3-1/2	5.0	.11
Ethanol	5%	200	3-1/2	6.0	.13
Sodium Methoxide	5%	200	3-1/2	30.0	.69
NaOH	3%	200	3-1/2	10.0	.22
KI (c)	5%	200	3-1/2	89.0	2.02
KI (d)	5%	200	3-1/2	30.0	.69
Na ₂ SO ₃	5%	200	3-1/2	215.0	4.9
Na ₂ CO ₃	100%	(e)	3-1/2	-----) No measur-
NaHCO ₃	100%	(e)	3-1/2	-----) able re-
NaHCO ₃ (f)	100%	(e)	3-1/2	-----) action

- (a) In 1% NaOH solution
- (b) 3% on NH₃ basis
- (c) In 1% KOH solution
- (d) In 1% HCl solution
- (e) 100 grams powder
- (f) Ansul NaHCO₃ dry powder

TABLE 5
OF₂ DECONTAMINATION STUDY
SPRAY CHAMBER TESTS

Decontaminant	SN	gm Ratio Spray:OF ₂	Spray		Spent Liquor Recovered			Note
			Rate ml/min	Time Sec.	ml	Fluoride gm	% OF ₂ Neutralized	
H ₂ O	1A	15:1	75	54	67.5	35.0	.004	0.13
"	1B	15:1	75	54	67.5	54.0	.005	0.16
"	2A	45:1	75	162	202.5	172.0	.95	30.0
"	2B	45:1	75	162	202.5	172.5	.0075	0.24
NH ₄ OH (5%)	3A	15:1	50	81	67.5	45.0	.625	19.6
"	3B	15:1	50	81	67.5	50.0	.125	3.94
"	4A	30:1	50	162	135	113.0	.50	15.75
"	4B	30:1	50	162	135	112.0	.66	20.8
"	5A	45:1	40	243	202.5	177.0	.65	20.7
"	5B	45:1	50	242	202.5	175.0	.93	29.3
"	6A	15:1	50	81	67.5	56.0	.14	4.41
"	6B	15:1	50	81	67.5	60.0	.36	11.32
"	7A	15:1	75	54	67.5	36.0	.19	5.98
"	7B	15:1	75	54	67.5	50.5	.31	9.76
"	7C	15:1	75	54	67.5	55.0	.48	15.10
"	7D	15:1	75	54	67.5	57.0	.58	18.26
"	7E	15:1	75	54	67.5	63.0	.50	15.70
"	8A	30:1	75	108	135	137.0	.97	30.5
"	8B	30:1	75	108	135	130.0	.70	22.0
"	9A	45:1	75	162	202.5	270.0	.77	24.2
"	9B	45:1	75	162	202.5	195.0	.88	27.7
"	9C	45:1	75	162	202.5	202.0	.75	23.6

TABLE 5 (CONTINUED)
OF₂ DECONTAMINATION STUDY
SPRAY CHAMBER TESTS

Decontaminant	SN	gm Ratio Spray:OF ₂	Spray			Spent Liquor Recovered			Note
			Rate ml/min	Time Sec.	ml	ml	gm Fluoride	% OF ₂ Neutralized	
NH ₄ OH(5%)	10A	15:1	100	41	67.5	52	.28	8.83	
"	10B	15:1	100	41	67.5	72	.42	13.20	
"	10C	15:1	100	41	67.5	65	.44	13.83	
"	11A	30:1	100	81	135	148	.68	21.4	
"	12A					27.0	.0625	1.97	c
"	12B					79.5	.375	11.8	c
"	12C					48.0	.313	9.88	c
"	12D					78.0	.250	7.89	c
"	12E					118.0	.500	15.8	c
"	12A-E	90:1	75	324	405	350.5	1.500	47.4	c
"	13A	45:1	750	17	202.5	180.0	.70	22.1	d
"	13B	45:1	750	17	202.5	190.0	.63	19.9	
"	14A	90:1	750	32.4	405.0	354.0	.76	24.0	
"	14B						.388	12.3	e
"	14A-B						1.148	36.3	f
"	14C	90:1	750	32.4	405	365.2	.097	30.6	
"	14D						.465	14.7	e
"	14C-D						1.435	45.3	f
NH ₄ OH(7 1/2%)	15A	90:1	750	32.4	405	367.0	.63	19.9	
"	15B	90:1	750	32.4	405	367.0	.94	29.7	
KOH (5%)	16A	90:1	750	32.4	405	350.0	.25	7.89	
"	16B	90:1	750	32.4	405	355.0	.31	9.79	

TABLE 5 (CONTINUED)
OF₂ DECONTAMINATION STUDY
SPRAY CHAMBER TESTS

Decontaminant	SN	gm Ratio Spray:OF ₂	Spray		Spent Liquor Recovered			Note
			Rate ml/min	Time Sec.	ml	Fluoride gm	% OF ₂ Neutralized	
Na ₂ SO ₃ (5%)	17A	90:1	750	32.4	405	355.0	1.04	32.8
"	17B	90:1	750	32.4	405	352.0	.62	19.6
Ethanol (5%)	18A	90:1	750	32.4	405	365.0	.0074	0.23
"	18B	90:1	750	32.4	405	360.0	.0074	0.23
Sodium Methoxide 5%	19A	90:1	750	32.4	405	360	.31	9.79
"	19B	90:1	750	32.4	405	---	---	---
5% KI in 1% KOH	20A	90:1	750	32.4	405	360	.62	19.6
"	20B	90:1	750	32.4	405	357	1.88	59.4
"	20C	>90:1	750	---	---	450	1.88	59.4
5% Na ₂ SO ₃ in 1% NaOH	21A	90:1	750	32.4	405	365	1.56	49.3
"	21B	90:1	750	32.4	405	365	1.56	49.3
5% Triethanolamine	22A	90:1	750	32.4	405	362	.73	23.1
"	22B	90:1	750	32.4	405	367	.83	26.2
5% Na ₂ SO ₃ in 1% NaOH	23A	90:1	375	65.0	405	391	1.41	44.5
"	23B	---	---	---	---	---	.125	3.95
"	23A-B	90:1	375	65.0	405	380	1.535	48.5
"	23C	90:1	375	65.0	405	380	1.34	42.3
"	23D	---	---	---	---	---	.188	5.94
"	23C-D	---	---	---	---	---	1.528	48.2

NOTE: In each run 4.5 gms of OF₂ was used.

a Runs 1 through 12 made using Spraying Systems Co. No. 5500X-1 Spray Nozzle.

b Flash seen when test tube was broken.

TABLE 5 (CONTINUED)
OF₂ DECONTAMINATION STUDY
SPRAY CHAMBER TESTS

c	Samples of spent liquor collected at one minute intervals after spray was started.
d	12E includes final drainage.
e	Runs 13 through 23 made using Spraying Systems Co. No. 1/8 G.G.S.S.-1 Fulljet Spray Nozzle.
f	Chamber rinsed out with water after spent liquor was removed to obtain residue fluorides.
g	Result combines spent liquor and rinse water.
h	Test tube failed to break and spent liquor not analyzed. Since other run showed low pickup, duplicate was not rerun.
i	Stopcock. leaked and some OF ₂ was lost when test tube was broken.
	Test tube was broken after spray started. Spray timing sequence was interrupted.
	Spray ratio higher than planned.

TABLE 6
OF₂ DECONTAMINANTS
FINAL ECONOMIC EVALUATION

<u>Material</u>	<u>Est. Cost Per Ton</u>	<u>Approx. Effect</u>	<u>Est. Cost to Neutralize 100 Lbs. OF₂</u>
NH ₄ OH	\$45.00	47%	\$ 215
Na ₂ SO ₃ in 1% NaOH	80.00 + 20.80	48%	473
KI in 1% KOH	2300.00 + 28.00	59%	8,878

NOTE: Above based on tests at a 90:1 decontaminant spray to OF₂ ratio. Decontaminant per se is 5% of total spray. Water costs are equal for each spray and were not considered in this evaluation.

TABLE 7
CANDIDATE MATERIALS
OF 2 COMPATIBILITY TESTS

<u>Manufacturer & Material</u>	<u>Fabricator</u>	<u>Status</u> *
1. Firestone Synthetic Rubber & Latex Company A. Butaprene RM-618	Raybestos-Manhattan	1
2. Dow Corning Corporation A. Silastic 50) B. Silastic LS-63) catalyzed with Luperco CST C. Silastic 950-U)	Dow Corning Corp.	1 1 1
3. Monsanto Chemical Co., Plastics Div. A. Lustran I 710 B. Cycolac ABS	Cadillac Plastic & Chemical Company	3 1
4. Marbon Chemical Company A. Cycolac H B. Cycolac L C. Cycolac LL D. Blendex 301		2 2 2 2
5. Goodyear Tire & Rubber Co. A. Budene B. Chemigum	Goodyear Tire & Rubber Co.	2 2
6. Rohm & Haas Company A. Acrylate (Butyl) Plastic	Rohm & Haas Company	2
7. Goodrich-Gulf Chemicals, Inc. A. Ameripol 34 B. Ameripol CB-220	Goodrich-Gulf	3 1
8. Borden Chemical Company A. Acrylon BA-12 B. Acrylon EA-5		3 3
9. Thiokol Chemical Company A. Thiokol ST B. Thiokol FA	Laurel Company	1 3
10. Shell Chemical Company A. Shell Polyethylene		2
11. Enjay Chemical Company A. Enjay EPR 404 B. Enjay Butyl 268 C. Enjay Butyl HT-10-66	Enjay Chemical Co.	1 1 1
12. Allied Chemical Corporation A. Aclar 22A B. Aclar 22C C. Aclar 33C D. Halon TFE G-80(H) E. Halon TFE G-80(L) F. Capran 77C G. Plaskon 8200 H. Plaskon 2200 I. Halon TFE G-80 J. Halon TFE G-50	Allied Chemical Corp.	1 1 1 1 1 1 1 1 1 1

TABLE 7
(CONTINUED)

<u>Manufacturer & Material</u>	<u>Fabricator</u>	<u>Status</u> [*]
13. duPont Chemical Company		
A. Teflon 5	Allied Chemical Corp.	1
B. Teflon 7	Allied Chemical Corp.	1
C. Viton 7250	Acme Hamilton Mfg. Corp.	1
D. Neoprene	Acme Hamilton Mfg. Corp.	2
E. Adiprene	Acme Hamilton Mfg. Corp.	2
F. FEP	Almac Plastics of N.J.	1
G. TFE	Almac Plastics of N.J.	1
H. Nylon 31 (Zytel)	Almac Plastics of N.J.	1
I. Nylon 105 (Zytel)	Almac Plastics of N.J.	1
14. Mobay Chemical Company	Eng'g. Block Sales Div. of Marbon Chemical	
A. Texin 480A		1
B. Texin 355D		1
C. Texin 192A		1
15. General Electric, Chem. Div.	Westlake Plastics	
A. Lexan Polycarbonate		1
16. General Electric, Silicone Prod. Department	General Electric Silicone Product Department	
A. SE-5211)		1
B. SE-555) Silicone Rubber		1
C. SE-5701)		1
17. Naugatuck Chemical		
A. Kralastic W)) ABS Resins	2
B. Paracrils		2
18. Celanese Polymer Company		
A. Fortiflex Polyethylenes		4
19. B. F. Goodrich Chemical Company	B.F. Goodrich Chem. Co.	
A. Abson 89015		2
B. Estane Polyurethane 58013		4
20. 3M Company, Chemical Division		
A. Kel-F 81	Fluocarbon Company	1
B. Kel-F Elastomer	Indus. Electronic Rubber Co.	1
C. CTFE	Almac Plastics	1
21. Union Carbide, Silicones Div.	Union Carbide, Silicones Div.	
A. Silicone Rubber K-1205(Red)		1
B. Silicon Rubber KW-1920(Neutral)		1
22. Pennsalt Chemical Company	The Fluocarbon Company	
A. Kynar		1

TABLE 7
(CONTINUED)

<u>Manufacturer & Material</u>	<u>Fabricator</u>	<u>Status</u> [*]
23. Dow Chemical Company		
A. High Impact Polypropylene		2
B. High Impact Styron		2
C. Tyril		2
D. Ethafoam		2
E. Polyethylene		2
F. Pelaspan		2
G. Pelaspan - Pac		2
H. Ethocel		2
24. Raybestos-Manhattan		
A. Silicone Rubber RM-66	Phoenix Asbestos Company	1
25. Connecticut Hard Rubber Company		
A. Viton 985	Mercer Rubber Company	1

Status^{*}:

1. Subjected to preliminary screening.
2. Manufacturer advised unsuitable.
3. Not available for testing.
4. Sample not received.

TABLE 8
MATERIAL COMPATIBILITY IN OF₂
PRELIMINARY SCREENING

Material	Test Results			
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Texin 355D	Sur. Degr.	None	-----	-----
Texin 480A	Sur. Degr.	None	-----	-----
Texin 192A	Sur. Degr.	None	-----	-----
R/M 618	None	None	None	None
R/M 66	None	None	None	None
Silastic 50	None	None	None	None
Silastic 950U	Sur. Degr.	None	-----	-----
Silastic LS-63	None	None	None	None
Thiokol ST Elastomer	None	None	None	Exploded
Enjay Butyl 268	None	None	Exploded	-----
Butyl HT-10-66	None	None	None	None
EPR 404	None	None	None	None
Aclar 22A	None	None	None	None
Capran 77C	None	None	None	None
Halon TFE G-50	None	None	None	None
Halon TFE G-80	None	None	None	None
Plaskon 8200	None	None	None	None
Plaskon 2200	None	None	None	None
Teflon 5	None	None	None	None
Teflon 7	None	None	None	None
SE 555	Sur. Degr.	None	-----	-----
SE 5211	Sur. Degr.	None	-----	-----
SE 5701	Sur. Degr.	None	-----	-----
Viton 7250	None	None	None	None
FEP	None	None	None	None
TFE (duPont)	None	None	None	None
CTFE	None	None	None	None
Nylon 31	None	None	None	None
Nylon 105	None	None	None	None
Kel-F 81	None	None	None	None
KW-1920	None	None	None	None
K-1205	Sur. Degr.	None	-----	-----
Kynar	None	None	None	None
Aclar 22C	None	None	None	None
Aclar 33C	None	None	None	None

TABLE 8
(CONTINUED)

<u>Material</u>	<u>Test Results</u>			
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Capran 77C *	Surface Tacky	----	-----	-----
Viton 985	None	None	None	None
Halon TFE G-80(H)	None	None	None	None
Halon TFE G-80(L)	None	None	None	None

NOTE: "None" indicates specimen remained unchanged.

"Sur. Degr." indicates degradation of specimen surface.

Test 1 - Gaseous OF₂.

Test 2 - Liquid N₂ (to evaluate suitability at cryogenic temperatures).

Test 3 and 4 - Liquid OF₂.

*Test 1 was repeated for ½ hour exposure

TABLE 9
MATERIAL COMPATIBILITY
24 HOUR OF₂ GAS EXPOSURE

Run No.	Material	Grams			Hardness		Observations
		Wt. 1	Wt. 2	Wt. 3	Before	After	
1	TFE	11.8517	11.8632	11.8518	58	58 (b)	Unchanged
	FEP	12.4255	12.4308	12.4229	60	60 (b)	Unchanged
	CTFE	12.0004	12.0006	12.0000	78	78 (b)	Unchanged
	Kynar	10.1628	10.1631	10.1613	78	78 (b)	Unchanged
	Zytel 105	6.6824	6.7133	-----	--	-----	Surface glossy and tacky
2	Silastic LS 63	17.4123	17.5801	17.3808	50	75 (a)	Bleached and hardened
	Silastic 50	13.1129	13.4303	12.7977	40	55 (a)	Bleached and hardened
	Zytel 31	4.8445	4.9532	-----	--	-----	Glossy and very tacky
3	K-1920	3.7298	-----	-----	25	---(a)	Completely disintegrated
	EPR 404	12.5694	-----	-----	61	---(a)	Severely oxidized
	Viton 7250	11.0790	-----	-----	65	---(a)	Severely oxidized
	HT-10-66	12.2941	-----	-----	68	---(a)	Severely oxidized
4	Viton 7250	11.1740	11.3682	11.1757	65	65 (a)	Unchanged
	EPR 404	12.5538	-----	-----	61	---(a)	Surface very sticky
	HT-10-66	12.6971	-----	-----	68	---(a)	Surface very sticky
5	Halon TFE G-80	11.7345	11.7381	11.7335	59	59 (b)	Unchanged
	Halon TFE G-50	11.4717	11.4731	11.4716	57	57 (b)	Unchanged
	Teflon 5	11.5835	11.5837	11.5834	58	58 (b)	Unchanged
	Teflon 7	12.3430	12.3431	12.3430	58	58 (b)	Unchanged
8	Plaskon 8200	7.6100	(d)	-----	82	---(b)	Surface glossy and tacky (d)
	R/M 66	3.1280	(e)	-----	60	78 (a)	Embrittled and Harder (e)
	Plaskon 2200	12.2029	12.1955	12.1954	78	78 (b)	Unchanged
10	R/M 618	7.0693	7.0765	7.0479	65	65 (a)	Unchanged

TABLE 9
(CONTINUED)

Run No.	Material	Grams			Hardness		Observations
		Wt. 1	Wt. 2	Wt. 3	Before	After	
11	CTFE (3M)	13.4257	13.4256	13.4242	78	78 (b)	Unchanged
	Viton 985	10.9069	10.9377	10.9248	74	74 (a)	Unchanged
13	Aclar 22A	.9706	.9690	.9690	(c)	-----	Unchanged
	Aclar 22C	.4847	.4808	.4808	(c)	-----	Unchanged
	Aclar 33C	.4657	.4617	.4615	(c)	-----	Unchanged

(a) Shore "A" Durometer.

(b) Shore "D" Durometer.

(c) Thin film, durometer readings not taken.

(d) Material stuck to bomb and supports and therefore could not be re-weighed with any degree of accuracy.

(e) Surface chipped off and specimen fragmented when bent.

NOTE: Wt. 1 - Specimen before OF₂ exposure

Wt. 2 - Specimen after OF₂ exposure

Wt. 3 - Exposed specimens after 22 hours at 75°C. in a vacuum oven

TABLE 10
MATERIAL COMPATIBILITY
7 DAY GASEOUS OF₂ EXPOSURE

Run No.	Material	Grams			Hardness		Observations
		Wt. 1	Wt. 2	Wt. 3	Before	After	
6	Halon G-80	11.8010	11.8017	11.7996	59	59	Unchanged
	Halon G-80	11.5839	11.5846	11.5804	59	59	Unchanged
	Halon G-50	11.6255	11.6280	11.6253	57	57	Unchanged
	Halon G-50	11.1958	11.1968	11.1958	57	57	Unchanged
	Teflon 5	11.6505	11.6509	11.6500	58	58	Unchanged
	Teflon 5	11.1385	11.1390	11.1380	58	58	Unchanged
	Teflon 7	12.3766	12.3779	12.3758	58	58	Unchanged
	Teflon 7	12.3341	12.3357	12.3326	58	58	Unchanged
7	TFE (DuPont)	12.1768	12.1810	12.1766	58	58	Unchanged
	TFE (DuPont)	12.2238	12.2278	12.2236	58	58	Unchanged
	FEP (DuPont)	12.5455	12.5521	12.5453	60	60	Unchanged
	FEP (DuPont)	12.5420	12.5516	12.5408	60	60	Unchanged
	CTFE (DuPont)	12.0123	12.0160	12.0129	78	78	Unchanged
	CTFE (DuPont)	12.4225	12.4244	12.4230	78	78	Unchanged
	Kynar (Pennsalt)	10.9656	10.9658	10.9634	78	78	Unchanged
	Kynar (Pennsalt)	11.1628	11.1597	11.1583	78	78	Unchanged
9	Plaskon 2200	12.1178	12.1063	12.1053	78	78	Unchanged
	Plaskon 2200	12.1810	12.1799	12.1789	78	78	Unchanged
	Viton 7250	11.1035	11.2113	11.1860	65	65 (a)	Unchanged
	Viton 7250	10.9166	11.0230	10.9998	65	65 (a)	Unchanged

TABLE 10
(CONTINUED)

Run No.	Material	Grams			Hardness		Observations
		Wt. 1	Wt. 2	Wt. 3	Before	After	
12	CTFE (3M)	13.2764	13.2766	13.2753	78	78	Unchanged
	CTFE (3M)	13.6016	13.6022	13.6016	78	78	Unchanged
	Viton 985	10.9516	10.9334	10.9192	74	74 (a)	Unchanged
	Viton 985	10.9753	10.9571	10.9439	74	74 (a)	Unchanged
	R/M 618	7.0472	7.0694	7.0552	65	65 (a)	Unchanged
	R/M 618	7.2770	7.2997	7.2844	65	65 (a)	Unchanged
14	Aclar 22A	.9674	.9680	.9674	(Film)	(Film)	Unchanged
	Aclar 22A	.9910	.9965	.9959	(Film)	(Film)	Unchanged
	Aclar 22C	.4782	.4786	.4776	(Film)	(Film)	Unchanged
	Aclar 22C	.4790	.4805	.4791	(Film)	(Film)	Unchanged
	Aclar 33C	.4659	.4660	.4657	(Film)	(Film)	Unchanged
	Aclar 33C	.4727	.4728	.4710	(Film)	(Film)	Unchanged

(a) Shore "A" Durometer. All other measurements made with a Shore "D" Durometer.

NOTE: Wt. 1 - Specimen before OF₂ exposure

Wt. 2 - Specimen after OF₂ exposure

Wt. 3 - Exposed specimens after 22 hours at 75°C. in a vacuum oven

TABLE 11
MATERIAL COMPATIBILITY
48 HOUR LIQUID OF₂ EXPOSURE

Material	G R A M S			Hardness	
	Wt. 1	Wt. 2	Wt. 3	Before	After
TFE	12.0376	12.0393	12.0372	58 ²	58
TFE	11.9779	11.9844	11.9778	58 ²	58
TFE	12.0913	12.0949	12.0912	58 ²	58
CTFE	12.4600	12.4626	12.4608	78 ²	78
CTFE	12.4389	12.4431	12.4392	78 ²	78
CTFE	12.0734	12.0754	12.0740	78 ²	78
FEP	13.0110	13.0143	13.0120	60 ²	60
FEP	12.9603	12.9635	12.9609	60 ²	60
FEP	12.6670	12.6719	12.6680	60 ²	60
VITON 7250	10.9860	10.9876	10.9825	65 ¹	65
VITON 7250	10.8060	10.7884	10.7816	65 ¹	65
VITON 7250	11.1382	11.1384	11.1336	65 ¹	65
VITON 985	10.8088	10.8087	10.8001	74 ¹	74
VITON 985	10.8361	10.8360	10.8264	74 ¹	74
VITON 985	10.7989	10.7989	10.7917	74 ¹	74
VITON 985	10.8279	10.8273	10.8184	74 ¹	74
RM 618	7.0921	7.0954	7.0641	65 ¹	65
RM 618	7.0766	7.0801	7.0492	65 ¹	65
RM 618	7.2810	7.2852	7.2545	65 ¹	65
RM 618	7.2800	7.2842	7.2564	65 ¹	65
HALON TFE G-50	11.0500	11.0500	11.0449	57 ²	57
HALON TFE G-50	11.6260	11.6255	11.6255	57 ²	57
HALON TFE G-50	11.0466	11.0466	11.0466	57 ²	57
HALON TFE G-50	11.7390	11.7390	11.7383	57 ²	57

TABLE 11 (CONTINUED)
MATERIAL COMPATIBILITY
48 HOUR LIQUID OF₂ EXPOSURE

Material	G R A M S			Hardness	
	Wt. 1	Wt. 2	Wt. 3	Before	After
TEFLON 5	11.7069	11.6950	11.6949	58 ²	58
TEFLON 5	11.5523	11.5487	11.5486	58 ²	58
TEFLON 5	11.8100	11.8049	11.8050	58 ²	58
TEFLON 5	11.5614	11.5541	11.5544	58 ²	58
HALON TFE G-80	11.5199	11.5181	11.5179	59 ²	59
HALON TFE G-80	11.7814	11.7805	11.7805	59 ²	59
HALON TFE G-80	11.6446	11.6369	11.6358	59 ²	59
HALON TFE G-80	12.2193	12.2192	12.2188	59 ²	59
TEFLON 7	12.3060	12.3059	12.3054	58 ²	58
TEFLON 7	12.1530	12.1529	12.1528	58 ²	58
TEFLON 7	12.4717	12.4704	12.4702	58 ²	58
TEFLON 7	12.2865	12.2858	12.2838	58 ²	58
ACLAR 22A	.9876	.9884	.9874	--	--
ACLAR 22A	.9805	.9809	.9805	--	--
ACLAR 22A	.9792	.9794	.9793	--	--
ACLAR 22A	.9891	.9891	.9891	--	--
HALON TFE 10 G-80 (L)	9494	10.9498	10.9490	58 ²	58
HALON TFE G-80 (L)	11.1625	11.1630	11.1629	58 ²	58
" (L)	10.4438	10.4438	10.4430	58 ²	58
" (L)	11.0296	11.0294	11.0290	58 ²	58
" (H)	10.6680	10.6689	10.6676	59 ²	59
" (H)	11.8687	11.8695	11.8684	59 ²	59
" (H)	11.6009	11.5994	11.5994	59 ²	59
" (H)	10.9563	10.9570	10.9570	59 ²	59

1 = Shore "A"; 2 = Shore "D"; Wt. 1 and 2 are weights before and after 48 hour exposure. Wt. 3 after exposed specimen had been heated in a vacuum oven at 75°C for 22 hours.

TABLE 12
MATERIAL COMPATIBILITY
7 DAY LIQUID OF₂ EXPOSURE

Material	*	G R A M S			Hardness	
		Wt. 1	Wt. 2	Wt. 3	Before	After
HALON TFE						
G-80 (L)	10.9235	10.9239	10.9251	10.9237	58 ²	58
"	10.7744	10.7746	10.7744	10.7744	58 ²	58
"	11.3939	11.3944	11.3959	11.3940	58 ²	58
"	11.0303	11.0311	11.0312	11.0309	58 ²	58
"	10.8804	10.8810	10.8815	10.8808	58 ²	58
"	10.6534	10.6538	10.6542	10.6540	58 ²	58
" (H)	11.1747	11.1756	11.1768	11.1752	59 ²	59
"	11.6116	11.6121	11.6130	11.6120	59 ²	59
"	11.2572	11.2577	11.2585	11.2578	59 ²	59
"	10.5113	10.5119	10.5120	10.5115	59 ²	59
"	11.5828	11.5831	11.5837	11.5829	59 ²	59
"	10.9469	10.9474	10.9478	10.9476	59 ²	59
HALON TFE						
G-80		12.4479	12.4500	12.4491	59 ²	59
HALON "		12.6209	12.6234	12.6223	59 ²	59
HALON "		12.6025	12.6050	12.6038	59 ²	59
HALON "		12.6091	12.6120	12.6109	59 ²	59
HALON TFE G-50		11.5460	11.5455	11.5449	57 ²	57
HALON "		12.1093	12.1110	12.1100	57 ²	57
HALON "		11.1312	11.1331	11.1325	57 ²	57
HALON "		11.8270	11.8295	11.8285	57 ²	57
TEFLON 7		12.4670	12.4691	12.4680	58 ²	58
TEFLON 7		12.3369	12.3393	12.3380	58 ²	58
TEFLON 7		12.2534	12.2557	12.2548	58 ²	58
TEFLON 7		12.2552	12.2578	12.2566	58 ²	58
TEFLON 5		11.5789	11.5810	11.5800	58 ²	58
TEFLON 5		12.0081	12.0106	12.0091	58 ²	58
TEFLON 5		12.0898	12.0916	12.0906	58 ²	58
TEFLON 5		11.3454	11.3476	11.3467	58 ²	58

TABLE 12 (CONTINUED)
MATERIAL COMPATIBILITY
7 DAY LIQUID OF₂ EXPOSURE

Material	*	G R A M S			Hardness	
		Wt. 1	Wt. 2	Wt. 3	Before	After
VITON 7250		11.0728	11.0725	11.0721	65 ¹	65
VITON 7250		10.9890	10.9894	10.9890	65 ¹	65
VITON 7250		11.2009	11.2015	11.2010	65 ¹	65
VITON 7250		11.1213	11.1217	11.1212	65 ¹	65
CTFE		11.2598	11.2598	11.2597	78 ²	78
CTFE		10.8616	10.8622	10.8620	78 ²	78
CTFE		11.3964	11.3741	11.3765	78 ²	78
CTFE		11.3241	11.3169	11.3168	78 ²	78
FEP		12.4702	12.4526	12.4552	60 ²	60
FEP		12.5042	12.5054	12.5092	60 ²	60
FEP		12.6762	12.6733	12.6747	60 ²	60
FEP		13.0635	13.0364	13.0370	60 ²	60
TFE		11.9304	11.9288	11.9279	58 ²	58
TFE		12.2321	12.2334	12.2332	58 ²	58
TFE		12.1023	12.1031	12.1024	58 ²	58
VITON 985		10.7667	10.7689	10.7595	74 ¹	74
VITON 985		10.8228	10.8248	10.8145	74 ¹	74
VITON 985		10.8709	10.8728	10.8625	74 ¹	74
VITON 985		10.9382	10.9398	10.9296	74 ¹	74
RM 618		7.3070	7.3185	7.2733	65 ¹	65
RM 618		7.3384	7.3496	7.2903	65 ¹	65
RM 618		7.3097	7.3212	7.2495	65 ¹	65
RM 618		7.3092	7.3220	7.2632	65 ¹	65
PLASKON 2200		10.4132	10.4119	10.4107	78 ²	78
PLASKON 2200		12.4524	12.4518	12.4510	78 ²	78
PLASKON 2200		11.9540	11.9526	11.9512	78 ²	78
PLASKON 2200		12.2276	12.2262	12.2244	78 ²	78
ACLAR 22A		.9921	.9914	.9910	--	--
ACLAR 22A		.9824	.9825	.9820	--	--

1 = Shore "A" Durometer; 2 = Shore "D" Durometer; * Original weight before aborted 96 hour run. Wt. 1 and 2 are weights before and after 7 day exposure. Wt. 3 after exposed specimen had been heated in a vacuum oven at 75°C for 22 hours.

TABLE 13
MATERIAL COMPATIBILITY
TENSILE TESTS

MATERIAL	SN	TENSILE TEST °F	OF 2 DAYS	TEST PHASE	TENSILE PSI	ELONG %	WEIGHT 1	WEIGHT 2	WEIGHT 3	AREA in ²	MC ₂ in ²
TFE	B	73	0	---	3430	252	---	---	---	---	---
TFE	C	73	0	---	3460	255	---	---	---	---	---
TFE	1-1A	73	1	Gas	3480	250	11.8517	11.8632	11.8518	7.2	+0.01
TFE	7-28	73	7	Gas	3200	290	12.1768	12.1810	12.1766	7.3	0.00
TFE	7-29	73	7	Gas	3360	250	12.2238	12.2278	12.2236	7.3	0.00
TFE	1-1B	73	2	Liq	4250	300	12.0376	12.0393	12.0372	7.3	-0.03
TFE	1-2	73	2	Liq	3520	270	11.9779	11.9844	11.9778	7.3	-0.01
TFE	1-3	73	2	Liq	3300	249	12.0913	12.0949	12.0912	7.3	-0.01
TFE	7-99	73	7	Liq	3760	283	12.1023	12.1031	12.1024	7.2	0.00
TFE	(1)	-320	0	---	15,271	3	---	---	---	7.3	---
TFE	(2)	-320	0	---	15,432	4	---	---	---	7.3	---
TFE	7-97	-320	7	Liq	10,419	5	11.9304	11.9288	11.9279	7.2	-0.05
TFE	7-98	-320	7	Liq	10,219	6	12.2321	12.2334	12.2332	7.3	+0.02
FEP	A	73	0	---	3080	300	---	---	---	7.3	---
FEP	B	73	0	---	2870	275	---	---	---	7.3	---
FEP	1-2	73	1	Gas	3140	300	12.4255	12.4308	12.4229	7.3	-0.36
FEP	7-30	73	7	Gas	3110	290	12.5455	12.5521	12.5453	7.3	0.00
FEP	7-31	73	7	Gas	3040	280	12.5420	12.5516	12.5408	7.3	-0.02
FEP	1-7	73	2	Liq	2780	245	13.0110	13.0143	13.0120	7.3	+0.07
FEP	1-8	73	2	Liq	2770	250	12.9603	12.9635	12.9609	7.3	+0.04
FEP	1-9	73	2	Liq	3540	338	12.6670	12.6719	12.6680	7.3	+0.07
FEP	7-95	73	7	Liq	3300	325	12.6762	12.6733	12.6747	7.3	+0.03
FEP	7-96	73	7	Liq	3520	400	13.0365	13.0346	13.0370	7.3	+0.01
FEP	(3)	-320	0	---	18,760	5	---	---	---	---	---
FEP	(4)	-320	0	---	18,840	4	---	---	---	---	---
FEP	7-93	-320	7	Liq	12,844	5	12.4702	12.4526	12.4552	7.3	-0.29
FEP	7-94	-320	7	Liq	12,923	6	12.5043	12.5054	12.5092	7.3	+0.10
CTFE	(A)	73	0	---	6480	87	---	---	---	---	---
CTFE	(B)	73	0	---	5740	105	---	---	---	---	---
CTFE	1-3	73	1	Gas	7050	70	12.0004	12.0006	12.0000	7.2	-0.05
CTFE	7-32	73	7	Gas	7170	103	12.0123	12.0160	12.0129	7.2	-0.01
CTFE	7-33	73	7	Gas	7150	98	12.4225	12.4244	12.4230	7.3	+0.01

TABLE 13
MATERIAL COMPATIBILITY
TENSILE TESTS (CONTINUED)

MATERIAL	SN	TENSILE TEST °F	OF 2 DAYS	TEST PHASE	TENSILE PSI	ELONG %	WEIGHT 1	WEIGHT 2	WEIGHT 3	AREA in ²	MC in ² x DAY
CTFE	1-4	73	2	Liq	6100	73	12.4600	12.4626	12.4608	7.3	+0.05
CTFE	1-5	73	2	Liq	6070	69	12.4389	12.4431	12.4392	7.3	+0.02
CTFE	1-6	73	2	Liq	6040	72	12.0734	12.0754	12.0740	7.3	+0.04
CTFE	7-91	73	7	Liq	5280	100	11.3964	11.3741	11.3765	7.2	-0.39
CTFE	7-92	73	7	Liq	5030	112	11.3241	11.3169	11.3168	7.2	-0.15
CTFE	(5)	-320	0	---	22,870	4	---	---	---	---	---
CTFE	(6)	-320	0	---	23,250	4	---	---	---	---	---
CTFE	7-89	-320	7	Liq	15,467	7	11.2598	11.2597	11.2597	7.2	0.00
CTFE	7-90	-320	7	Liq	15,119	7	10.8616	10.8622	10.8620	7.2	+0.01
CTFE	11-44	73	1	Gas	7110	75	13.4257	13.4256	13.4242	7.4	-0.20
CTFE	11-45	73	1	Gas	6380	70	14.0410	14.0415	14.0389	7.4	-0.28
CTFE	12-48	73	7	Gas	5880	70	13.2764	13.2766	13.2753	7.3	-0.02
CTFE	12-49	73	7	Gas	5980	45	13.6016	13.6022	13.6016	7.3	0.00
Halon TFE G-80		73	0	---	4140	300	---	---	---	---	---
" G-80		73	0	---	4800	320	---	---	---	---	---
" G-80	5-16	73	1	Gas	4750	340	11.7345	11.7381	11.7335	7.2	-0.14
" G-80	6-20	73	7	Gas	4325	330	11.8010	11.8017	11.7996	7.2	-0.03
" G-80	6-21	73	7	Gas	4125	300	11.5839	11.5846	11.5804	7.2	-0.07
" G-80	3-25	73	2	Liq	4630	352	12.2193	12.2192	12.2188	7.2	-0.03
" G-80	3-26	73	2	Liq	4980	367	11.6446	11.6369	11.6358	7.2	-0.61
" G-80	6-63	73	7	Liq	4500	330	12.6025	12.6050	12.6038	7.3	+0.03
" G-80	6-64	73	7	Liq	4250	320	12.6091	12.6120	12.6109	7.3	+0.04
" G-80	(11)	-320	0	---	13,250	4	---	---	---	---	---
" G-80	(12)	-320	0	---	13,280	4	---	---	---	---	---
" G-80	3-27	-320	2	Liq			11.7814	11.7805	11.7805	7.2	-0.06
" G-80	3-28	-320	2	Liq			11.5199	11.5181	11.5179	7.2	-0.14
" G-80	6-61	-320	7	Liq	9365	7	12.4479	12.4500	12.4491	7.3	+0.02
" G-80	6-62	-320	7	Liq	9243	6	12.6209	12.6234	12.6223	7.3	+0.03
" G-80	(4)	-320	0	---	14,450	5	---	---	---	---	---
" G-80	(5)	-320	0	---	14,700	5	---	---	---	---	---
" G-80 (H)	1	73	0	---	3960	350	---	---	---	---	---
" G-80 (H)	2	73	0	---	3900	355	---	---	---	---	---

TABLE 13
MATERIAL COMPATIBILITY
TENSILE TESTS (CONTINUED)

MATERIAL	SN	TENSILE TEST °F	OF 2 DAYS	TEST PHASE	TENSILE PSI	ELONG %	WEIGHT 1	WEIGHT 2	WEIGHT 3	AREA in ²	MG in ² x DAY
HALON TFE G-80(H)	15-66	73	1	Gas	1837	572	11.2746	11.2794	11.2734	7.1	-0.17
" G-80 (H)	15-67	73	1	Gas	3960	430	10.6962	10.6984	10.6960	7.1	-0.03
" G-80 (H)	16-72	73	7	Gas	4010	393	10.5312	10.5370	10.5311	7.1	0.00
" G-80 (H)	16-73	73	7	Gas	3170	445	10.8969	10.9030	10.8967	7.1	0.00
" G-80 (H)	4-37	73	2	Liq	4060	367	10.9563	10.9570	10.9570	7.1	+0.05
" G-80 (H)	4-38	73	2	Liq	4380	379	11.6009	11.5994	11.5994	7.2	-0.11
" G-80 (H)	553	73	7	Liq	4280	340	11.6121	11.6130	11.6120	7.2	+0.01
" G-80 (H)	5-54	73	7	Liq	2660	550	11.1756	11.1768	11.1752	7.1	+0.01
" G-80 (H)	1C	-320	0	---	4360	2	---	---	---	---	---
" G-80 (H)	2C	-320	0	---	8330	4	---	---	---	---	---
" G-80 (H)	4-39	-320	2	Liq	---	---	11.8687	11.8695	11.8684	7.3	-0.02
" G-80 (H)	4-40	-320	2	Liq	---	---	10.6880	10.6689	10.6676	7.1	-0.03
" G-80 (H)	5-49	-320	7	Liq	6250	4	10.9474	10.9478	10.9476	7.1	+0.01
" G-80 (H)	5-50	-320	7	Liq	7606	3	11.5831	11.5837	11.5829	7.2	0.00
" G-80 (H)	5-51	-320	7	Liq	8608	7	10.5119	10.5120	10.5115	7.1	0.00
" G-80 (H)	5-52	-320	7	Liq	4594	3	11.2577	11.2585	11.2578	7.1	+0.01
HALON TFE G-80(L)	---	73	0	---	6380	450	---	---	---	---	---
" G-80 (L)	---	73	0	---	6430	450	---	---	---	---	---
" G-80 (L)	15-68	73	1	Gas	6320	452	11.0030	11.0050	11.0027	7.2	-0.04
" G-80 (L)	15-69	73	1	Gas	5620	392	11.1717	11.1755	11.1710	7.2	-0.10
" G-80 (L)	16-70	73	7	Gas	>5510*	423	11.6003	11.6073	11.6001	7.3	0.00
" G-80 (L)	16-71	73	7	Gas	5910	440	11.2846	11.2911	11.2843	7.2	-0.01
" G-80 (L)	4-41	73	2	Liq	6020	443	11.0296	11.0294	11.0290	7.1	-0.04
" G-80 (L)	4-42	73	2	Liq	5990	438	10.4438	10.4438	10.4430	7.1	-0.06
" G-80 (L)	5-59	73	7	Liq	6700	420	10.7746	10.7744	10.7744	7.2	0.00
" G-80 (L)	5-60	73	7	Liq	6060	380	10.9239	10.9251	10.9237	7.2	0.00
" G-80 (L)	3C	-320	0	---	11,000	7	---	---	---	---	---
" G-80 (L)	4C	-320	0	---	11,060	11	---	---	---	---	---
" G-80 (L)	8D	-320	0	---	17,550	6	---	---	---	---	---
" G-80 (L)	9D	-320	0	---	17,500	6	---	---	---	---	---
" G-80 (L)	4-43	-320	2	Liq	---	---	11.1625	11.1630	11.1629	7.1	+0.03

* Slipped out of grip; did not break at this point.

TABLE 13
MATERIAL COMPATIBILITY
TENSILE TESTS (CONTINUED)

MATERIAL	SN	TENSILE TEST °F	OF 2 DAYS	TEST PHASE	TENSILE PSI	ELONG %	WEIGHT 1	WEIGHT 2	WEIGHT 3	AREA in ²	MC ₂ in x DAY
HALON TFE G-80 (L)	4-44	-320	2	Liq			10.9494	10.9498	10.9490	7.1	-0.03
" G-80 (L)	5-55	-320	7	Liq	10,702	7	10.6538	10.5642	10.6540	7.1	+0.03
" G-80 (L)	5-56	-320	7	Liq	10,751	8	10.8810	10.8815	10.8808	7.2	+0.01
" G-80 (L)	5-57	-320	7	Liq	10,906	10	11.0311	11.0312	11.0309	7.2	+0.01
" G-80 (L)	5-58	-320	7	Liq	11,039	10	11.3944	11.3959	11.3940	7.2	0.00
HALON TFE G-50		73	0	---	2780	250	---	---	---	---	---
" G-50		73	0	---	2920	270	---	---	---	---	---
" G-50	5-17	73	1	Gas	3020	200	11.4717	11.4731	11.4716	7.2	-0.01
" G-50	6-22	73	7	Gas	2775	190	11.6225	11.6280	11.6253	7.2	0.00
" G-50	6-23	73	7	Gas	2420	150	11.1958	11.1968	11.1958	7.2	0.00
" G-50	3-33	73	2	Liq	3520	261	11.7390	11.7390	11.7383	7.2	-0.05
" G-50	3-34	73	2	Liq	3410	232	11.0466	11.0466	11.0466	7.2	0.00
" G-50	6-67	73	7	Liq	2830	205	11.1312	11.1331	11.1325	7.2	+0.03
" G-50	6-68	73	7	Liq	3160	225	11.8270	11.8295	11.8285	7.2	+0.03
" G-50	(9)	-320	0	---	8820	1	---	---	---	---	---
" G-50	(10)	-320	0	---	5780	1	---	---	---	---	---
" G-50	(2)D	-320	0	---	7700	2	---	---	---	---	---
" G-50	(3)D	-320	0	---	8340	2	---	---	---	---	---
" G-50	3-35	-320	2	Liq			11.6260	11.6255	11.6255	7.2	-0.03
" G-50	3-36	-320	2	Liq			11.0500	11.0500	11.0499	7.1	-0.01
" G-50	6-65	-320	7	Liq	4953	5	11.5460	11.5455	11.5449	7.3	-0.02
" G-50	6-66	-320	7	Liq	6290	7	12.1093	12.1110	12.1100	7.2	+0.01
TEFLON 5		73	0	---	3870	250	---	---	---	---	---
"		73	0	---	3640	250	---	---	---	---	---
"	5-18	73	1	Gas	3540	220	11.5835	11.5837	11.5834	7.2	-0.01
"	6-24	73	7	Gas	3810	260	11.6505	11.6509	11.6500	7.2	-0.01
"	6-25	73	7	Gas	3760	250	11.1385	11.1390	11.1380	7.2	-0.01
"	3-31	73	2	Liq	3500	208	11.5523	11.5487	11.5486	7.2	-0.26
"	3-32	73	2	Liq	3830	231	11.7069	11.6950	11.6949	7.2	-0.55
"	6-75	73	7	Liq	3940	230	12.0898	12.0916	12.0906	7.2	+0.02
"	6-76	73	7	Liq	3820	240	11.3454	11.3476	11.3467	7.2	+0.03
"	13	-320	0	---	12,410	3	---	---	---	---	---

TABLE 13
MATERIAL COMPATIBILITY
TENSILE TESTS (CONTINUED)

MATERIAL	SN	TENSILE TEST °F	OF ₂ DAYS	TEST PHASE	TENSILE PSI	ELONG %	WEIGHT 1	WEIGHT 2	WEIGHT 3	AREA in ²	MC ₂ in x DAY
TEFLON 5	14	-320	0	---	12,500	2	---	---	---	---	---
"	(6)D	-320	0	---	12,800	3	---	---	---	---	---
"	(7)D	-320	0	---	13,200	3	---	---	---	---	---
"	3-29	-320	2	Liq	13,150	3	11.5614	11.5541	11.5544	7.2	-0.48
"	3-30	-320	2	Liq	13,270	3	11.8100	11.8049	11.8050	7.2	-0.35
"	6-73	-320	7	Liq	8429	5	11.5739	11.5810	11.5800	7.2	+0.02
"	6-74	-320	7	Liq	8117	5	12.0081	12.0106	12.0091	7.2	+0.02
TEFLON 7		73	0	---	4850	350	---	---	---	---	---
"		73	0	---	4375	320	---	---	---	---	---
"	5-19	73	1	Gas	4660	330	12.3430	12.3431	12.3430	7.3	0.00
"	6-26	73	7	Gas	5060	350	12.3766	12.3779	12.3758	7.3	-0.02
"	6-27	73	7	Gas	5000	350	12.3341	12.3357	12.3326	7.3	-0.03
"	3-23	73	2	Liq	4740	339	12.1530	12.1529	12.1528	7.2	-0.01
"	3-24	73	2	Liq	4900	346	12.3060	12.3059	12.3054	7.2	-0.04
"	6-71	73	7	Liq	4900	336	12.2534	12.2557	12.2548	7.3	+0.03
"	6-72	73	7	Liq	4190	300	12.2552	12.2578	12.2566	7.3	+0.03
"	(15)	-320	0	---	13,370	5	---	---	---	---	---
"	(16)	-320	0	---	13,550	5	---	---	---	---	---
"	1D	-320	0	---	14,600	7	---	---	---	---	---
"	3-21	-320	2	Liq	14,700	6	12.2865	12.2858	12.2838	7.2	-0.19
"	3-22	-320	2	Liq	14,500	5	12.4712	12.4704	12.4702	7.2	-0.07
"	6-69	-320	7	Liq	9306	10	12.4670	12.4691	12.4680	7.3	+0.02
"	6-70	-320	7	Liq	9167	8	12.3369	12.3393	12.3380	7.2	+0.02
VITON 7250		73	0	---	2200	400	---	---	---	---	---
"		73	0	---	2360	500	---	---	---	---	---
"	4-13	73	1	Gas	2240	380	11.1740	11.3682	11.1757	7.3	+0.23
"	9-41	73	7	Gas	2120	510	11.1035	11.2113	11.1860	7.3	+1.61
"	9-42	73	7	Gas	2060	490	10.9166	11.0203	10.9998	7.3	+1.63
"	2-10	73	2	Liq	2190	425	10.9860	10.9876	10.9825	7.3	-0.24
"	2-11	73	2	Liq	2370	477	10.8060	10.7884	10.7816	7.3	-1.67
"	2-12	73	2	Liq	2400	470	11.1382	11.1384	11.1336	7.3	-0.31
"	7-87	73	7	Liq	2090	465	11.2009	11.2015	11.2010	7.3	0.00

TABLE 13
MATERIAL COMPATIBILITY
TENSILE TESTS (CONTINUED)

MATERIAL	SN	TENSILE TEST °F	OF 2 DAYS	TEST PHASE	TENSILE PSI	ELONG %	WEIGHT 1	WEIGHT 2	WEIGHT 3	AREA in ²	MG-2 in ² x DAY
VITON 7250	7-88	73	7	Liq	2250	505	11.1213	11.1217	11.1212	7.3	0.00
"		-320	0	---			---	---	---	---	
"		-320	0	---			---	---	---	---	
"	7-85	-320	7	Liq	11,270	5	11.0728	11.0725	11.0721	7.3	-0.01
"	7-86	-320	7	Liq	10,794	6	10.9890	10.9894	10.9890	7.3	0.00
"	11D	73	0	---	2365	483	---	---	---	---	
"	12D	73	0	---	2360	---	---	---	---	---	
VITON 985		73	0	---	1870	414	---	---	---	---	
"		73	0	---	1780	410	---	---	---	---	
"	11-46	73	1	Gas	1985	470	10.9069	10.9377	10.9248	7.3	+2.45
"	11-47	73	1	Gas	1985	472	10.9757	11.0064	10.9925	7.3	+2.31
"	12-50	73	7	Gas	1862	470	10.9516	10.9334	10.9192	7.3	-0.63
"	12-51	73	7	Gas	1862	472	10.9753	10.9571	10.9439	7.3	-0.57
"	2-13	73	2	Liq	1890	430	10.7989	10.7989	10.7917	7.3	-0.49
"	2-14	73	2	Liq	1980	483	10.8088	10.8087	10.8001	7.3	-0.59
"	2-15	73	2	Liq	2040	462	10.8361	10.8360	10.8264	7.3	-0.66
"	2-16	73	2	Liq	1950	434	10.8279	10.8273	10.8184	7.3	-0.65
"	8-79	73	7	Liq	1970	500	10.8709	10.8728	10.8625	7.3	-0.16
"	8-80	73	7	Liq	1875	480	10.9382	10.9398	10.9296	7.3	-0.17
"	5C	-320	0	---	7130	6	---	---	---	---	
"	6C	-320	0	---	7300	4	---	---	---	---	
"	8-77	-320	7	Liq			10.7667	10.7689	10.7595	7.3	-0.14
"	8-78	-320	7	Liq	9063	1	10.8228	10.8248	10.8145	7.3	-0.16
RM 618		73	0	---	1630	400	---	---	---	---	
"		73	0	---	1630	420	---	---	---	---	
"	10-43	73	1	Gas	1665	410	7.0693	7.0765	7.0479	7.2	-2.97
"	12-52	73	7	Gas	1645	445	7.0472	7.0694	7.0694	7.3	+0.16
"	12-53	73	7	Gas	1706	420	7.2770	7.2997	7.2844	7.3	+0.14
"	2-17	73	2	Liq	1620	399	7.0921	7.0954	7.0641	7.3	-1.92
"	2-18	73	2	Liq	1645	400	7.0766	7.0801	7.0492	7.3	-1.87
"	2-19	73	2	Liq	1690	410	7.2810	7.2852	7.2545	7.3	-1.81
"	2-20	73	2	Liq	1645	398	7.2800	7.2842	7.2564	7.3	-1.60

TABLE 13
MATERIAL COMPATIBILITY
TENSILE TESTS (CONTINUED)

MATERIAL	SN	TENSILE TEST °F	OF 2 DAYS	TEST PHASE	TENSILE PSI	ELONG %	WEIGHT 1	WEIGHT 2	WEIGHT 3	AREA in ²	MC ₂ in ² x DAY
RM 618		-320	0	BROKE IN CHOCK							
"		-320	0								
"	8-83	73	7	Liq	1610	433	7.3097	7.3212	7.2495	7.3	-1.18
"	8-84	73	7	Liq	1610	425	7.3092	7.3220	7.2632	7.3	-0.90
"	8-81	-320	7	Liq	7030	3	7.3070	7.3185	7.2733	7.3	-0.66
"	8-82	-320	7	Liq	4861	2	7.3384	7.3496	7.2903	7.3	-0.94
PLASKON 2200		-320	0								
"		73	0								
"		73	0								
"	8-38	73	1	Gas	6900	25	12.2029	12.1955	12.1954	7.2	-1.02
"	9-39	73	7	Gas	5900	27	12.1178	12.1063	12.1053	7.2	-0.25
"	9-40	73	7	Gas	6020	80	12.1810	12.1799	12.1789	7.2	-0.04
"	8-102	73	7	Liq	3920	20	11.9540	11.9526	11.9512	7.1	-0.06
"	8-103	73	7	Liq	4400	20	12.2276	12.2262	12.2244	7.1	-0.06
"	11C	-320	0		11,040	7					
"	12C	-320	0		11,060	4					
"	8-100	-320	7	Liq	11,592	9	10.4132	10.4119	10.4107	6.9	-0.05
"	8-101	-320	7	Liq	12,544	8	12.4524	12.4518	12.4510	7.1	-0.03
ACLAR 22A											
"		73	0								
"		73	0								
"	13-54	73	1	Gas			.9706	.9690	.9690	5.9	-0.27
"	13-55	73	1	Gas			.9631	.9610	.9607	5.9	-0.41
"	14-64	73	7	Gas			.9970	.9965	.9959	5.9	-0.03
"	14-65	73	7	Gas			.9674	.9680	.9674	5.9	0.00
"	4-45	73	2	Liq			.9891	.9891	.9891	5.9	0.00
"	4-46	73	2	Liq			.9792	.9794	.9793	5.9	+0.01
"	13C	-320	0		11,100	3					
"	14C	-320	0		10,000	4					
"	4-47	-320	2	Liq			.9805	.9809	.9805	5.9	0.00
"	4-48	-320	2	Liq			.9876	.9884	.9884	5.9	+0.07
"	8-104	-320	7	Liq	13,700	3	.9914	.9914	.9910	5.9	-0.03
"	8-105	-320	7	Liq	14,500	3	.9824	.9825	.9820	5.9	-0.01

NOTE: Material not subjected to 48 hour LOF₂ exposure.

NOTE: All weights in grams

TABLE 14
MATERIAL COMPATIBILITY
CRYSTALLINITY

<u>Material</u>	<u>SN</u>	<u>Method of Determination</u>		
		<u>Specific Gravity</u>	<u>Density Gradient</u>	<u>Infrared</u>
Halon TFE G-50	1A-3	45.7%		
Halon TFE G-50	1B-3	-----		
Halon TFE G-50	1C-3	50.1	46.5%	63%
Halon TFE G-50	1D-3	49.3	48.6	
Halon TFE G-50	2A-3	49.0		
Halon TFE G-50	2B-3	48.3		
Halon TFE G-50	2C-3	49.7	45.4	
Halon TFE G-50	2D-3	52.2	49.7	
Halon TFE G-80	3A-3	49.0		
Halon TFE G-80(H)	4A-3	52.5		
Halon TFE G-80(H)	4B-3	51.5		
Halon TFE G-80(H)	5A-3	52.5		
Halon TFE G-80(H)	5B-3	63.6		
Halon TFE G-80(L)	6A-3	40.0		
Halon TFE G-80(L)	6B-3	44.0		
Halon TFE G-80(L)	7A-3	43.6		
Halon TFE G-80(L)	7B-3	43.6		
TFE	8A-3	43.6		
TFE	8B-3	44.0		
TFE	8C-3	45.0	43.6	
TFE	8D-3	48.7	48.3	
Teflon 7	9A-3	49.3		
Teflon 5	10A-3	49.0		
Halon TFE G-50	1A-5	50.4	47.2	
Halon TFE G-50	1B-5	51.2	47.5	
Halon TFE G-50	2A-5	51.9	48.3	
Halon TFE G-50	2B-5	51.9	48.3	
Halon TFE G-80	3A-5	52.9	50.1	
Halon TFE G-80	3B-5	52.9	49.3	
Halon TFE G-80	4A-5	54.0	48.6	
Halon TFE G-80	4B-5	53.3	49.7	
Plaskon 2200			54	
CTFE			34	

TABLE 15
ORIFICE CALIBRATION
CTFE ORIFICE 0.0135" DIA.

INITIAL PRESSURE PSI GAGE	WATER DELIVERED Ft ³ /sec.	ORIFICE AREA Ft ²	VELOCITY THRU ORIFICE Ft/sec.	DISCHARGE COEFFICIENT C _d	REYNOLDS No IN ORIFICE Re
28	2.79x10 ⁻⁵	0.0999x10 ⁻⁵	27.9	0.435	2900
51	3.92x10 ⁻⁵	0.0999x10 ⁻⁵	39.2	0.450	4070
97.7	5.58x10 ⁻⁵	0.0999x10 ⁻⁵	55.9	0.464	5800
148	7.10x10 ⁻⁵	0.0999x10 ⁻⁵	71.1	0.482	7400
198	8.19x10 ⁻⁵	0.0999x10 ⁻⁵	82.0	0.477	8520
249	9.36x10 ⁻⁵	0.0999x10 ⁻⁵	93.7	0.489	9700
302	10.6x10 ⁻⁵	0.0999x10 ⁻⁵	106.0	0.502	11000
352	11.5x10 ⁻⁵	0.0999x10 ⁻⁵	115.1	0.502	12000
409	13.5x10 ⁻⁵	0.0999x10 ⁻⁵	135.1	0.545	14000

NOTE: Orifice calibrated with water at ambient temperature.

TABLE 16
LIQUID OF₂-
DYNAMIC TEST #1

<u>Run No.</u>	<u>PSIG</u>		<u>ft./sec.</u> <u>Velocity</u>	<u>Flow Rate</u> <u>lbs./sec.</u>	<u>Discharge</u>	<u>Reynolds #</u>
	<u>Initial</u>	<u>Final</u>			<u>Coefficient</u> <u>Cd</u>	<u>in Orifice</u> <u>Re</u>
1	25	24	21.5	0.0024	0.48	3970
2	56	54	33.0	0.0037	0.49	6100
3	108	106	46.5	0.0052	0.50	8600
4A	148	147				
4B	147	147	56.2	0.0063	0.51	10400
4C	147	147				
5	196	194	68.5	0.0076	0.54	12700
6	252	250	79.2	0.0089	0.55	14600
7	299	296	86.5	0.0097	0.55	16000
8	360	357	98.5	0.0111	0.56	18200
9	398	396	104	0.0116	0.57	19200
10	464	460	111	0.0124	0.57	20500
11	480	478	114	0.0128	0.57	21000

NOTE: 1. All runs were 5 seconds.
 2. Pressure downstream of the orifice is atmospheric.
 3. Test specimen, TFE made from duPont resin. Orifice
 0.0135" I.D.

TABLE 17
ANNIN VALVE
COMPONENT IDENTIFICATION

<u>No.</u>	<u>DESCRIPTION</u>	<u>MATERIAL</u>
1	Cap Screw - Valve Body	304 SS
2	Nut - Valve Body	303 SS
3	Washer - Spring Lock, Valve Body	304 SS
4	Adapter - Globe, Body	304 SS
5	Body - Valve	304 SS
6	Not applicable to this valve	
7	Seat Ring - Valve	Copper
8	Plug - Valve	304 SS
9	Half Ring - End Flange, Valve	304 SS
10	Flange - End, Valve	304 SS
11	Nut - Plug, Valve	303 SS
12	Tube Retainer Lower Guide	304 SS
13	Gasket - Bellows Seal	Copper
14	Gasket - Extension, Body	Aluminum
15	Extension - Body	304 SS
16	Assembly - Bellows	347 SS
17	Nut - Mtg., Gland Flange	Carbon Steel
18	Flange - Gland, Valve	Carbon Steel
19	Bolt - Mtg., Gland Flange	Carbon Steel
20	Extension - Stem	304 SS
21	Cap Screw - Mtg., Extension	304 SS
22	Half Ring - Flange, Body Extension	304 SS
23	Flange - Extension, Body	304 SS
24	Half Ring - Flange, Body	304 SS
25	Flange - Body	304 SS
26	Washer - Spring Lock, Body Extension	304 SS
27	Nut - Mtg., Body Extension	303 SS
28	Cap Screw - Valve Body	304 SS
29	Not applicable	
30	Not applicable	
31	Stem Guide - Lower	304 SS
32	Stem Guide - Upper	304 SS
33	Packing	Teflon
34	Packing Gland	304 SS

NOTE: Refer to FIGURE 11.

TABLE 18
ANNIN VALVE #1
COMPONENT WEIGHT LOSSES AND MEASUREMENTS

<u>Part</u>	<u>Material</u>	<u>Part weight in grams</u>		
		<u>Before</u>	<u>After</u>	<u>Loss</u>
Bellows	347 SS	467	383	84
Bellows Seal Gasket	Copper	6.6	---	6.6
Flange Gaskets (3)	Aluminum	7.5	2.0	5.5
Body Extension Gasket	Aluminum	.3	---	.3
Stem and Lock Nut	304 & 303 SS	62.5	41.6	29.9
Cap Screws-Mtg., Extension (4)	304 SS	170.0	167.0	3.0
Valve Body and Flanges	304 SS	2911	2861	50
Bellow Assembly Upper Flange	304 SS	422.3	412.5	<u>9.8</u>
		TOTAL	180.1	

Component Measurements

1. The valve plug at its shoulder had been reduced from .686" to .670".
2. The valve plug stem which is nominally .363" O.D. ranged from .272" to .337".
3. The stem guide (part 31) had been enlarged from .367" I.D. to .412" I.D.
4. The tube retainer lower guide (part 12) had been enlarged from .682" I.D. to .732" I.D.
5. Measurements of the burned out elbow showed no changes (.505" O.D.) indicating the rupture was definitely a burnout and not a pressure bursting.

TABLE 19
OF₂ BURNOUT INVESTIGATION
PRELIMINARY TESTS SUMMARY

Run No.	Initial PSIG	Final PSIG	Calories Input	Oxygen in Sample*	
				Method A	Method B
1	60	60	3.1	Not sampled	
2	200	200	3.1	<1.0%	1.34%
3	300	305	3.1	<1.0%	-----
4	400	410	3.1	<1.0%	2.12%
5	515	515	3.1	<1.0%	1.78%
6	600	600	3.1	-----	Sample lost
7	700	701	3.1	<1.0%	-----
8	800	805	3.1	<1.0%	-----
9	908	910	3.1	<1.0%	-----
10	1112	1119	3.1	<1.0%	-----
11	1503	1519	3.1	<1.0%	-----

*Oxygen resulting from OF₂ decomposition by Pyrofuze ignition

Method A - mass spectroscopy (mole per cent).

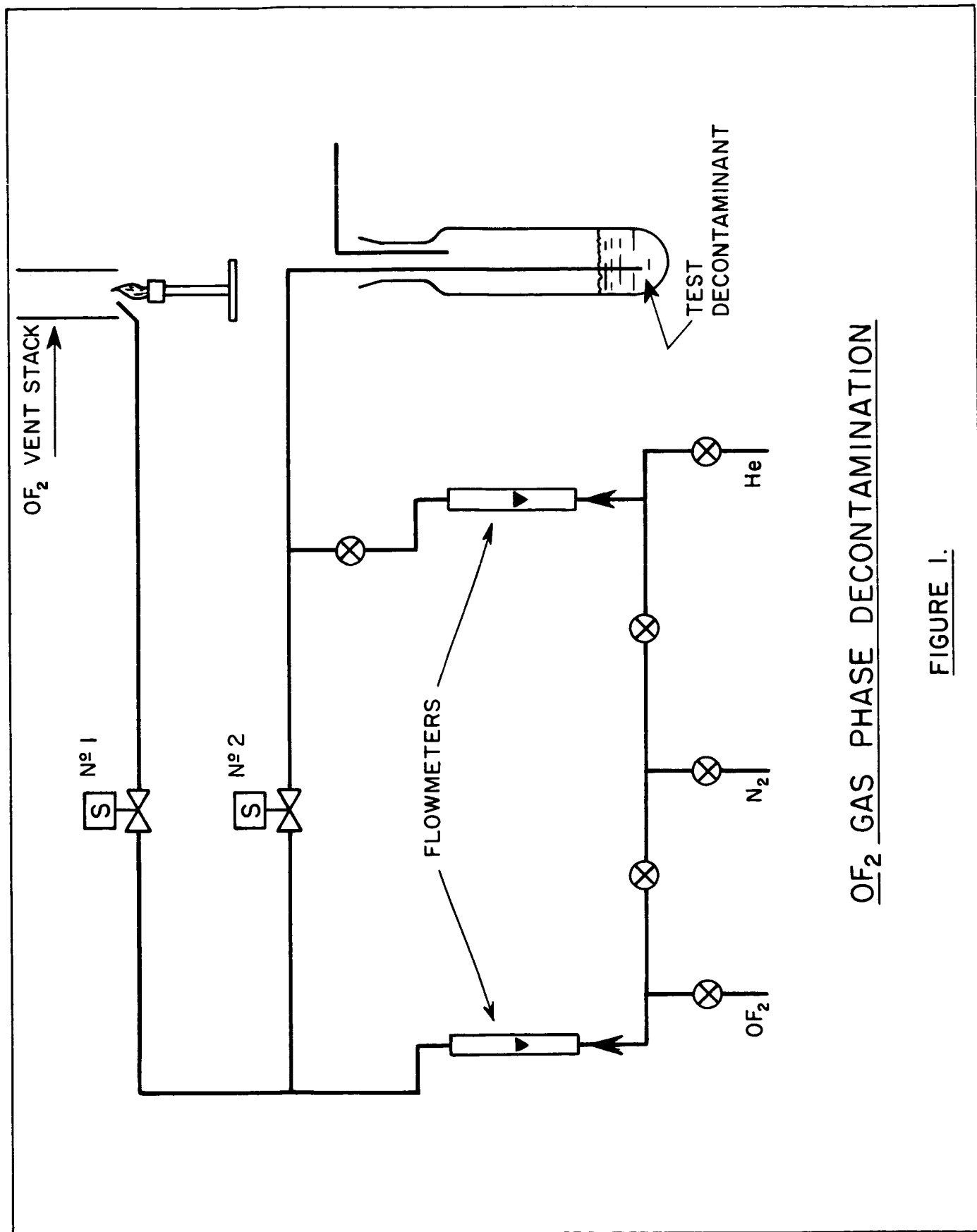
Method B - gas chromatograph (area per cent) includes O₂ and N₂ present. Column did not separate these gases.

TABLE 20
OF₂ BURNOUT INVESTIGATION
FINAL TESTS SUMMARY

<u>Run No.</u>	<u>PSIG</u>			<u>Calories</u> <u>Input</u>	<u>% Gas Composition*</u>			<u>% OF₂</u> <u>Decomposition</u>
	<u>Initial</u>	<u>Peak</u>	<u>Final</u>		<u>O₂</u>	<u>F₂</u>	<u>OF₂</u>	
12	300	375	318	35.5	15.4	20.7	63.9	32.5**
13	300	440	322	64.8	8.9	15.5	75.6	19.1
14	300	600	361	132.0	19.9	36.1	44.0	47.5
15	300	700	380	263.9	28.7	49.0	22.3	82.1
16	600	1300	865	264.9	34.3	64.8	.9	98.7

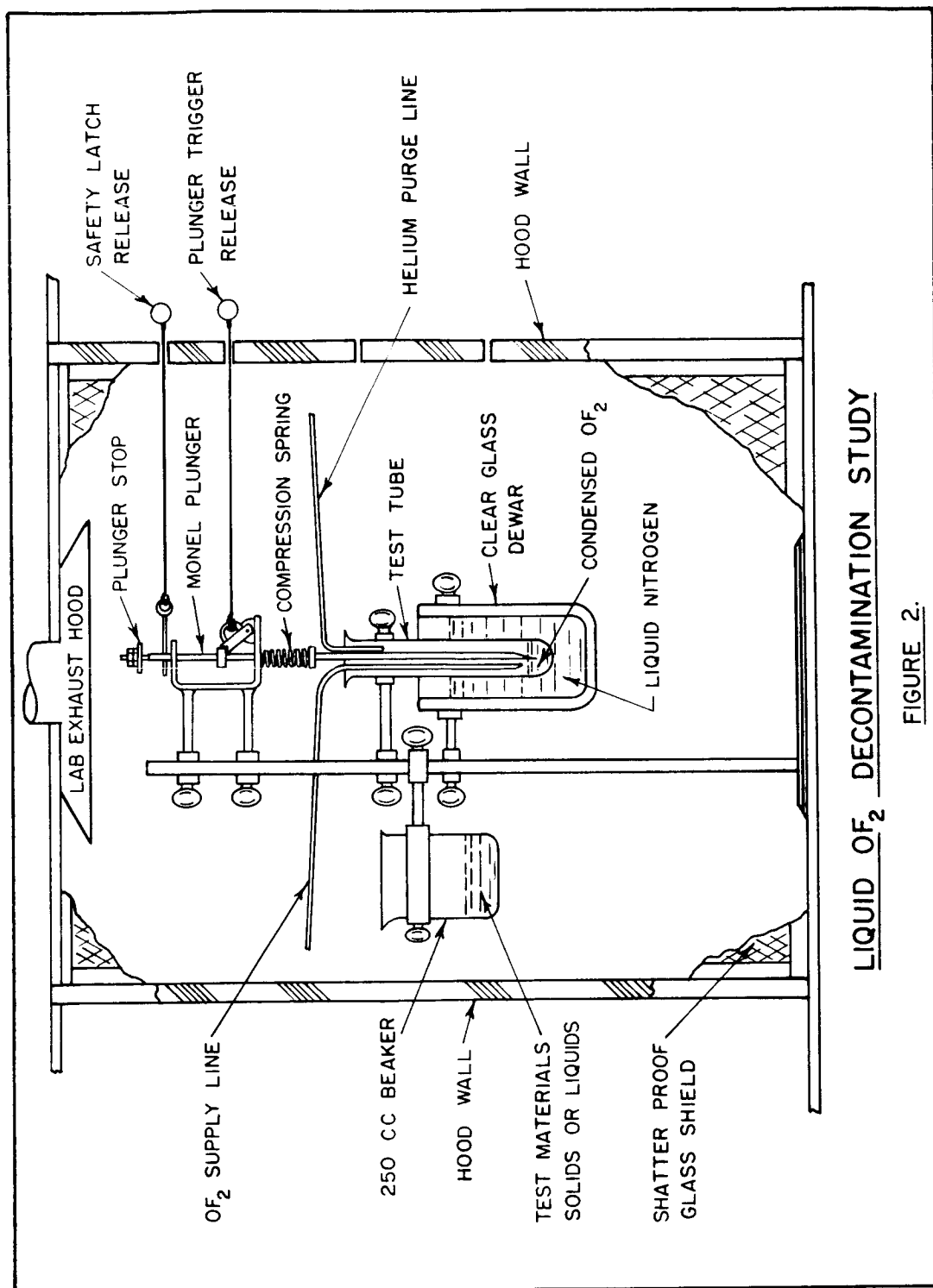
*The final gas composition after Pyrofuze ignition was calculated on the basis of the pressure increase in the reactor. These calculations included fluorine consumption from the OF₂-Pyrofuze reactions.

**In this run the electrodes were totally consumed as well as the Pyrofuze. The OF₂ consumption in the reactions with these metals was considered in calculating the final gas composition as well as the OF₂ decomposition.



OF_2 GAS PHASE DECONTAMINATION

FIGURE 1.



LIQUID OF₂ DECONTAMINATION STUDY

FIGURE 2.

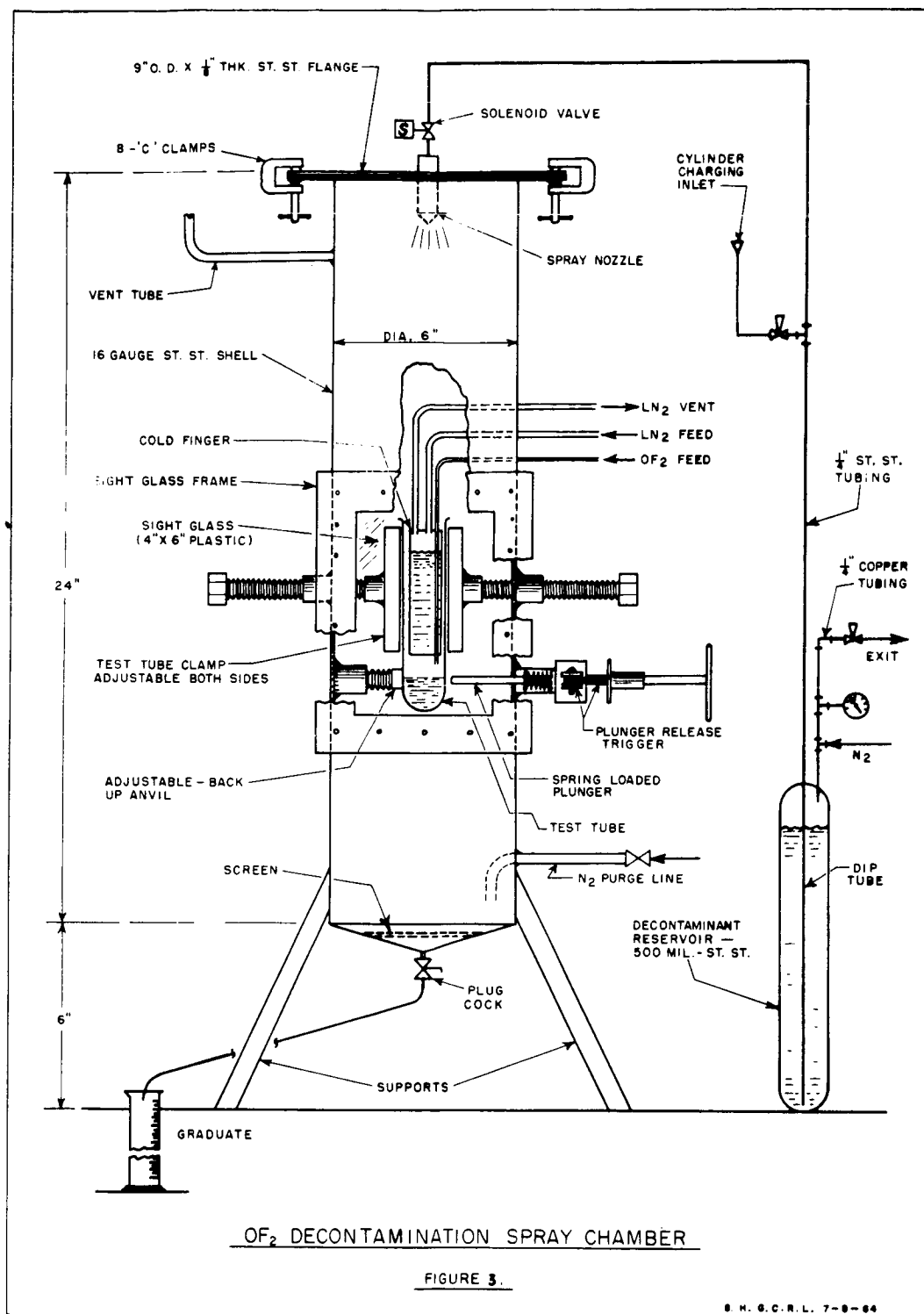
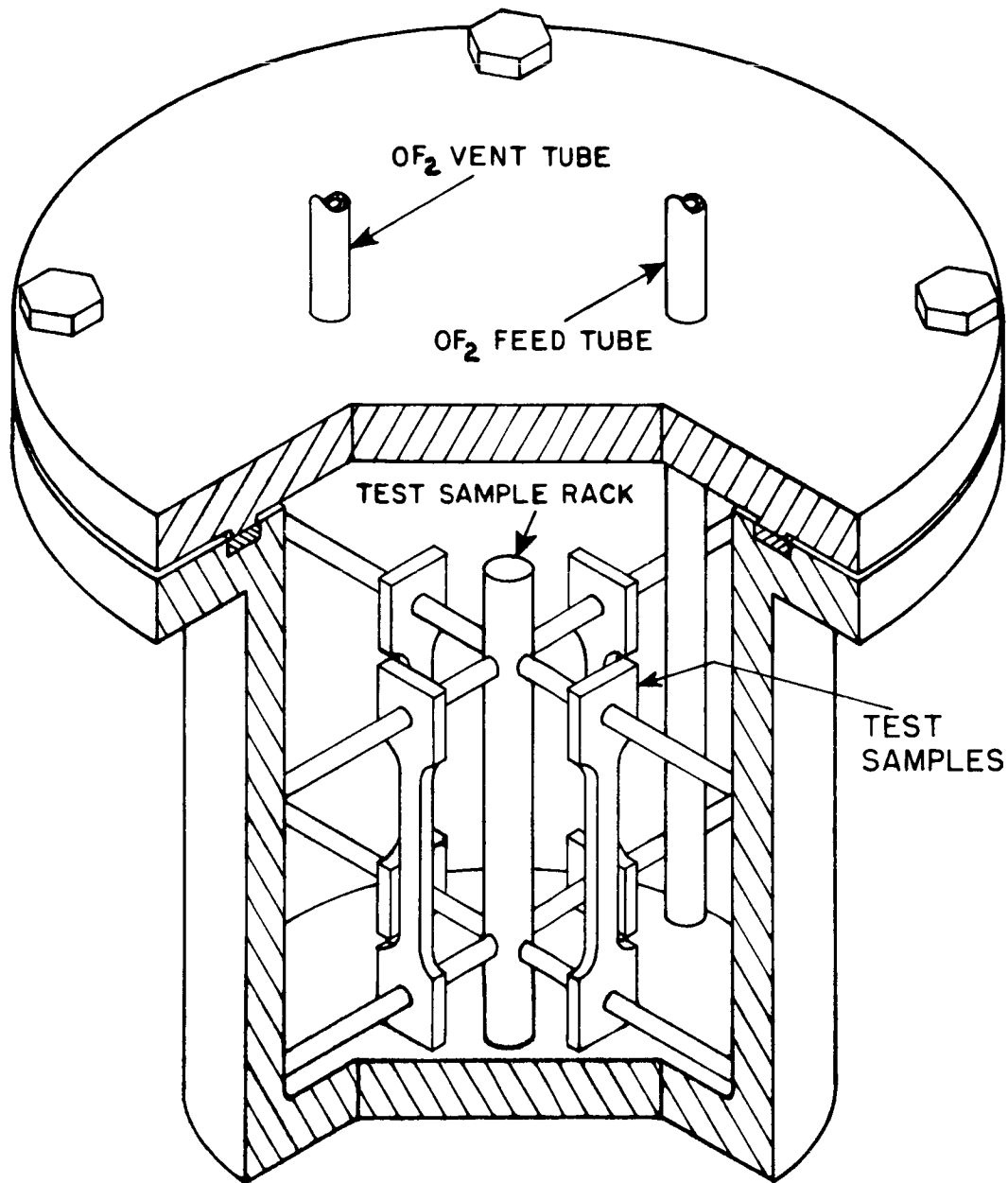


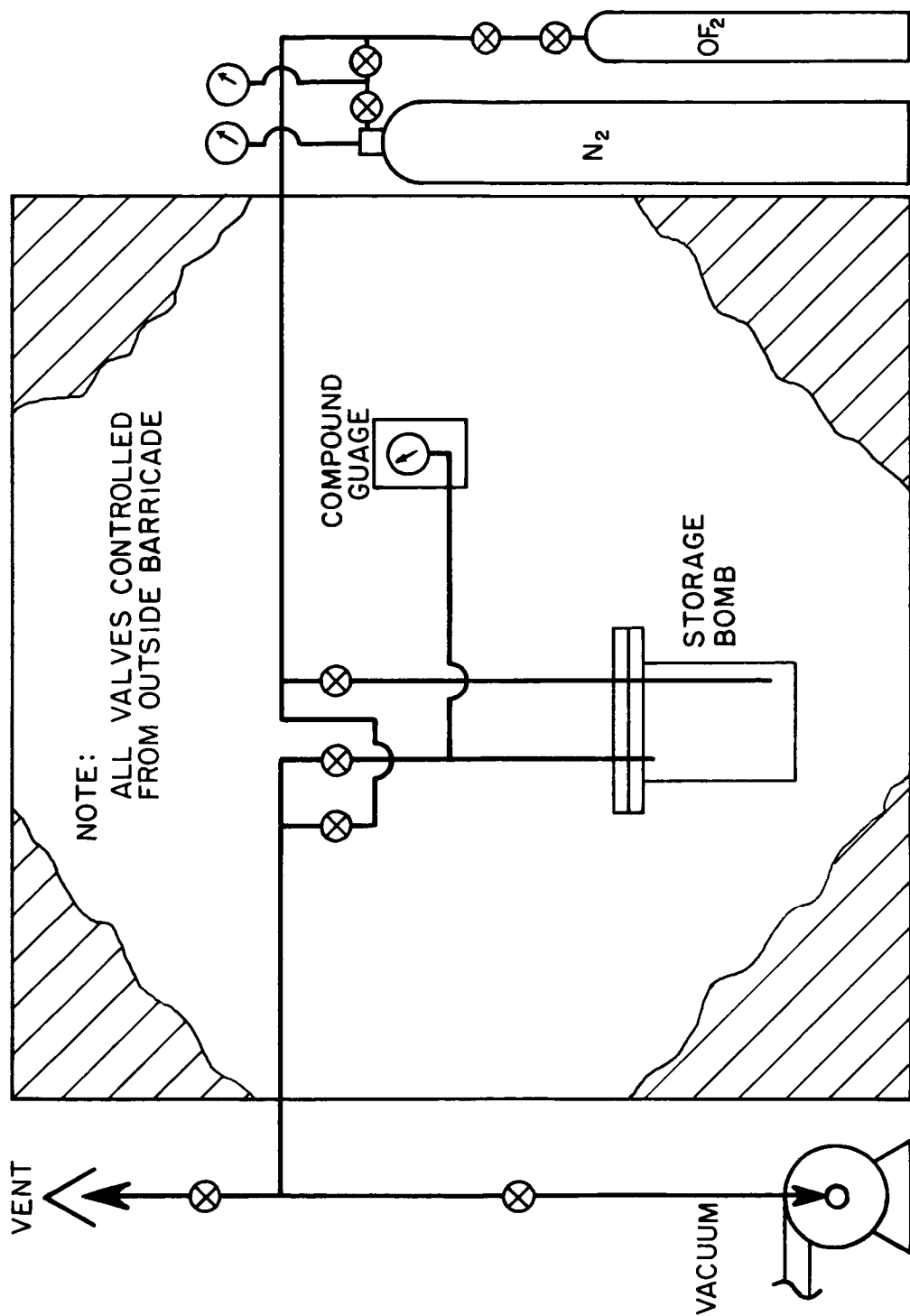
FIGURE 3.

S. H. G. C. R. L. 7-8-64



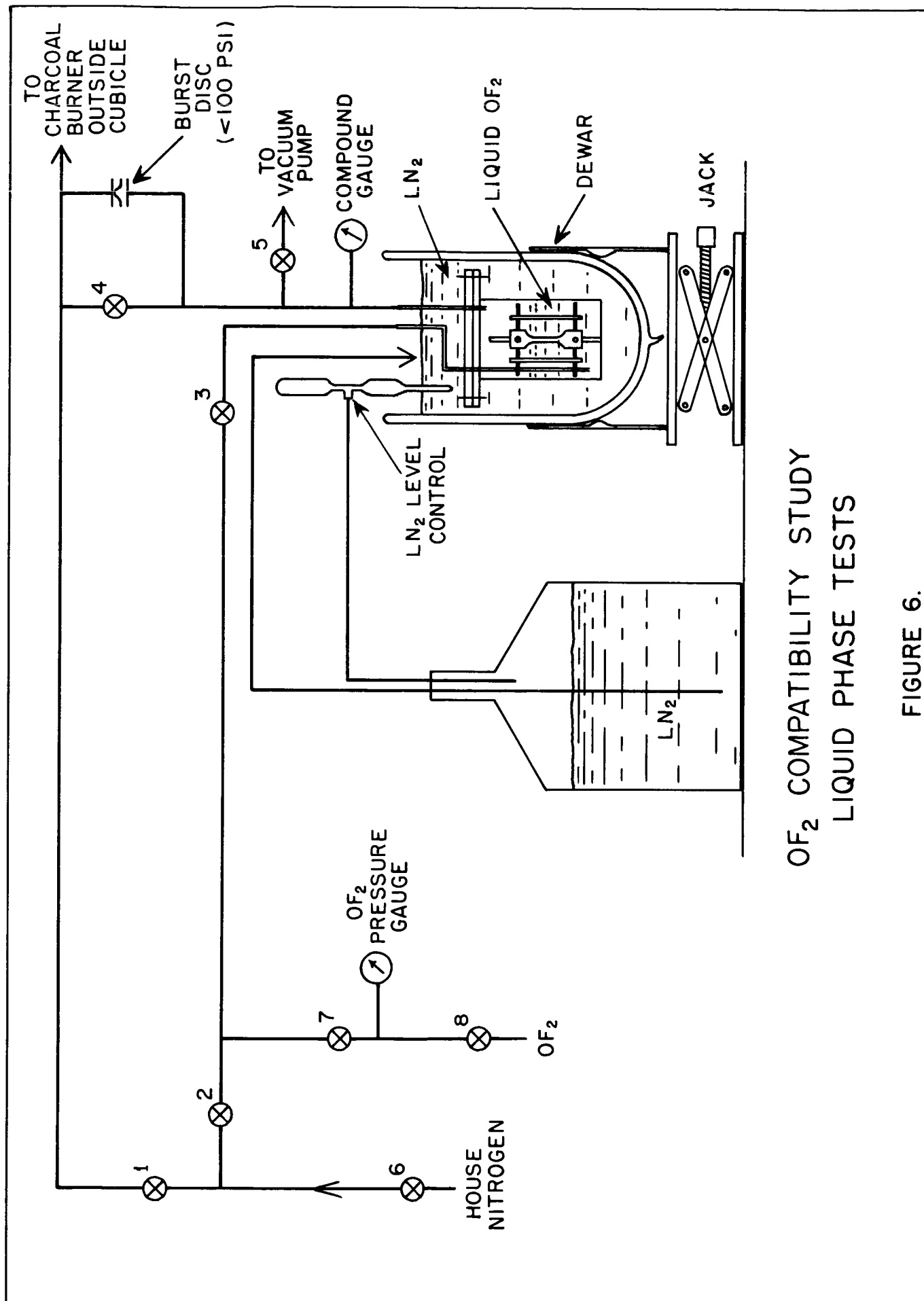
STORAGE TESTS OF₂ BOMB

FIGURE 4.



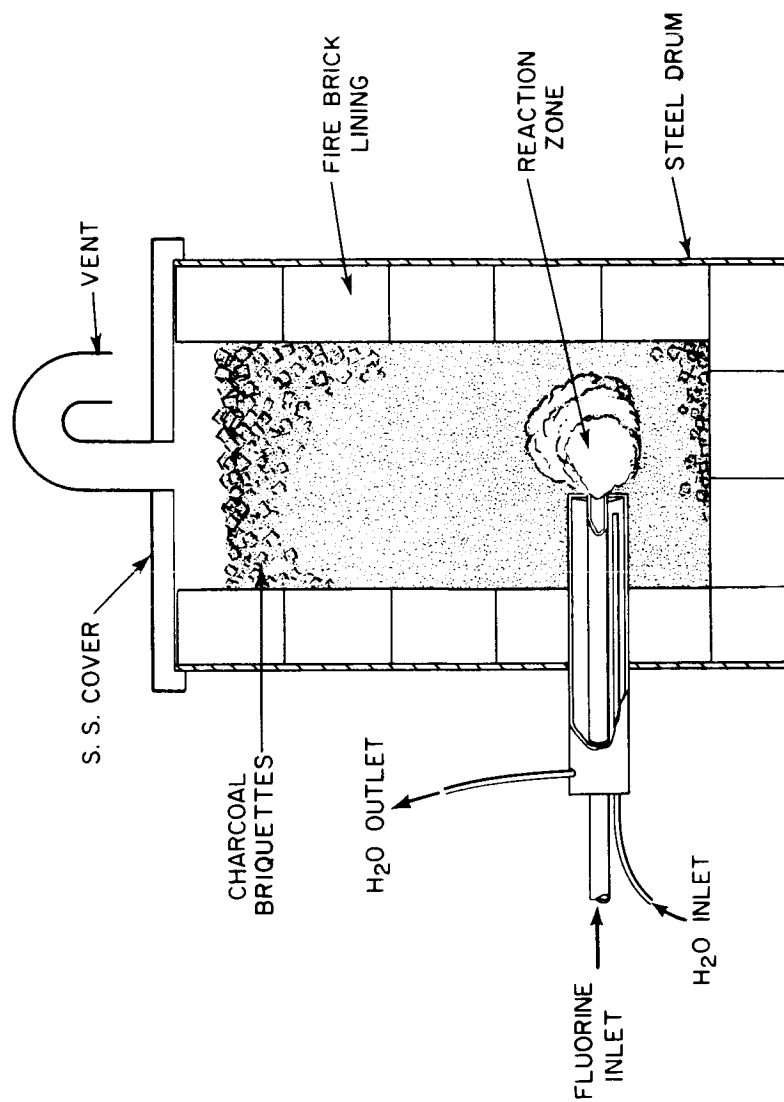
OF_2 COMPATABILITY STUDY
GAS PHASE TESTS

FIGURE 5.



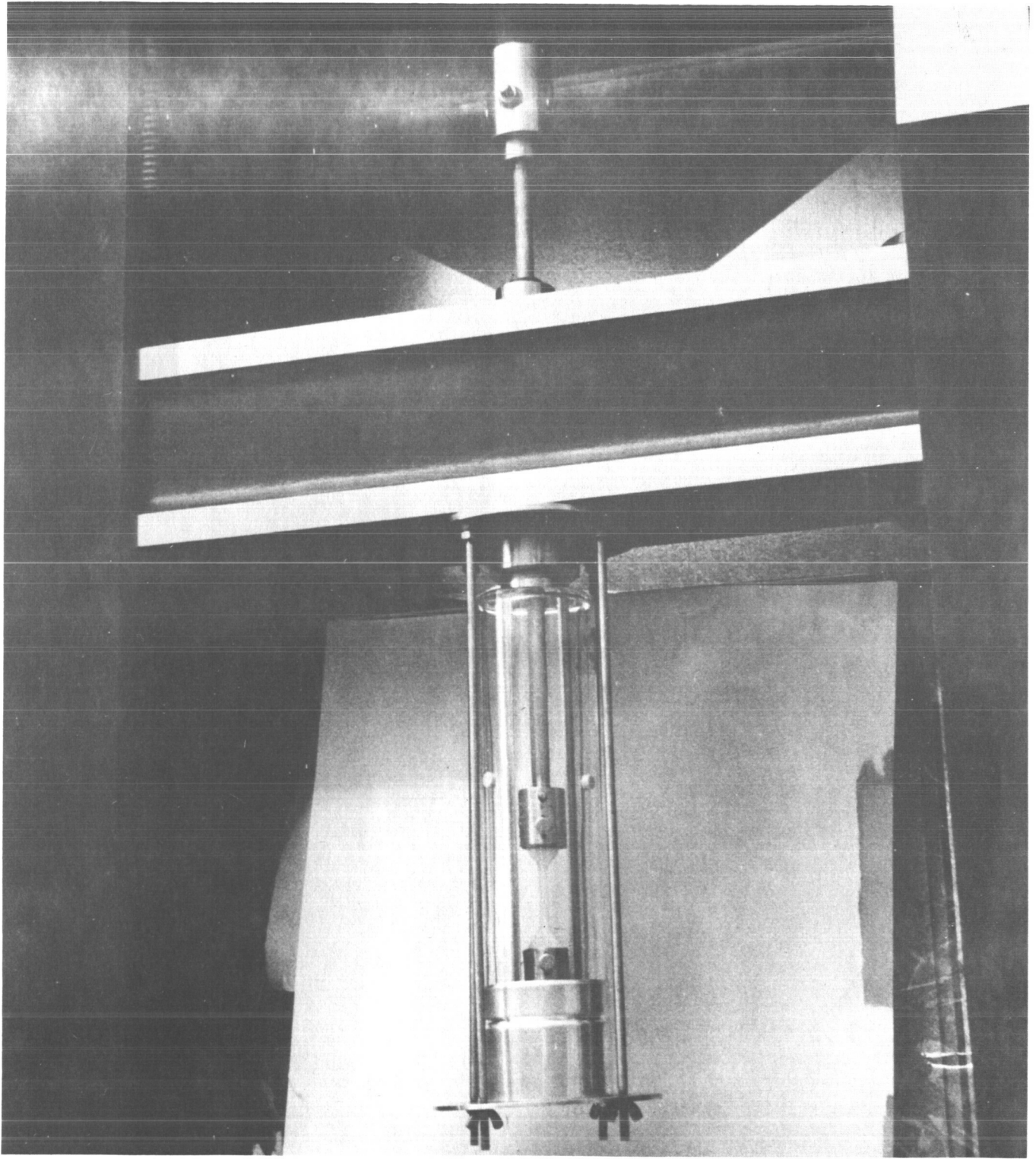
OF₂ COMPATIBILITY STUDY
LIQUID PHASE TESTS

FIGURE 6.

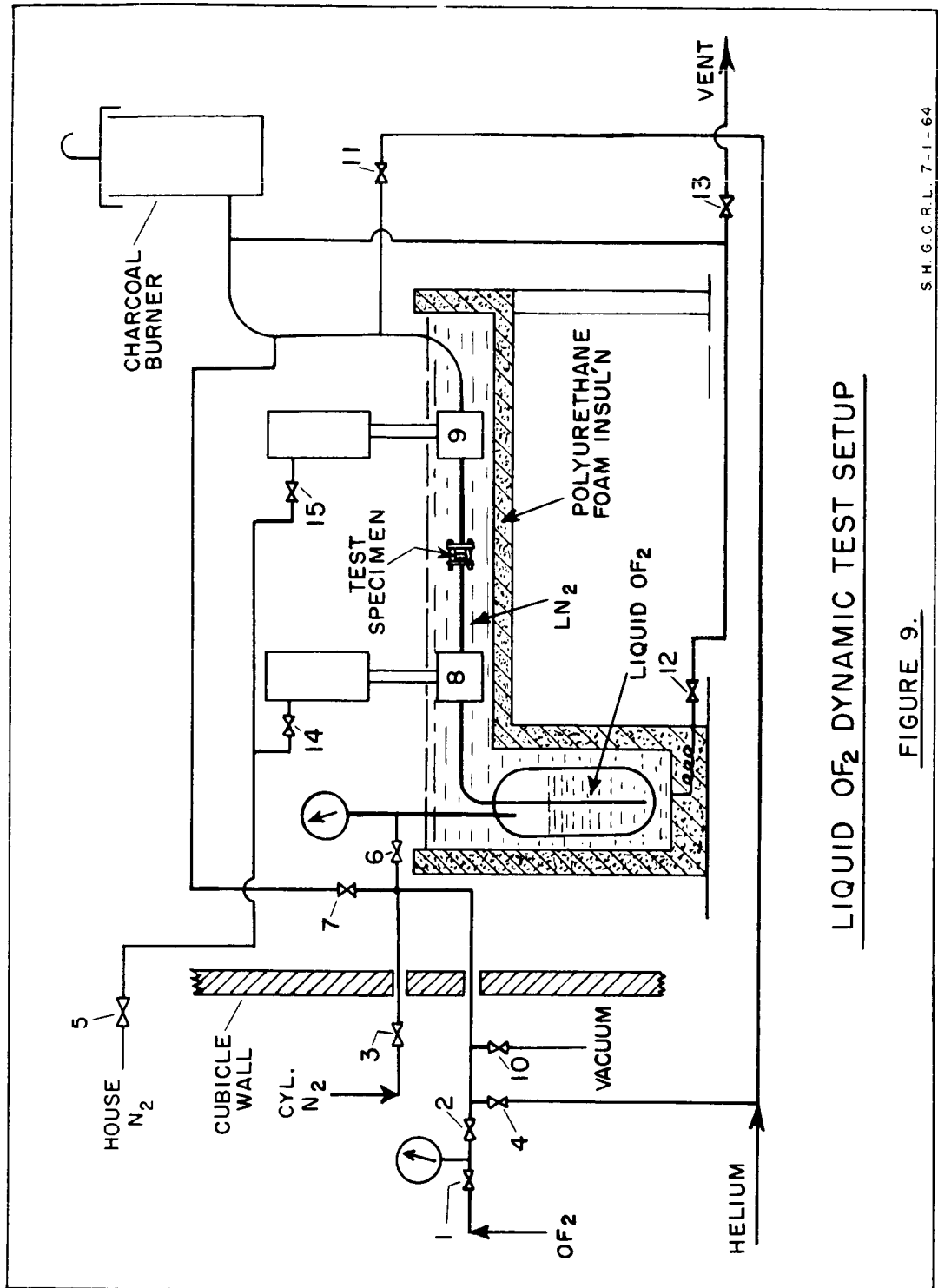


CHARCOAL BURNER

FIGURE 7.



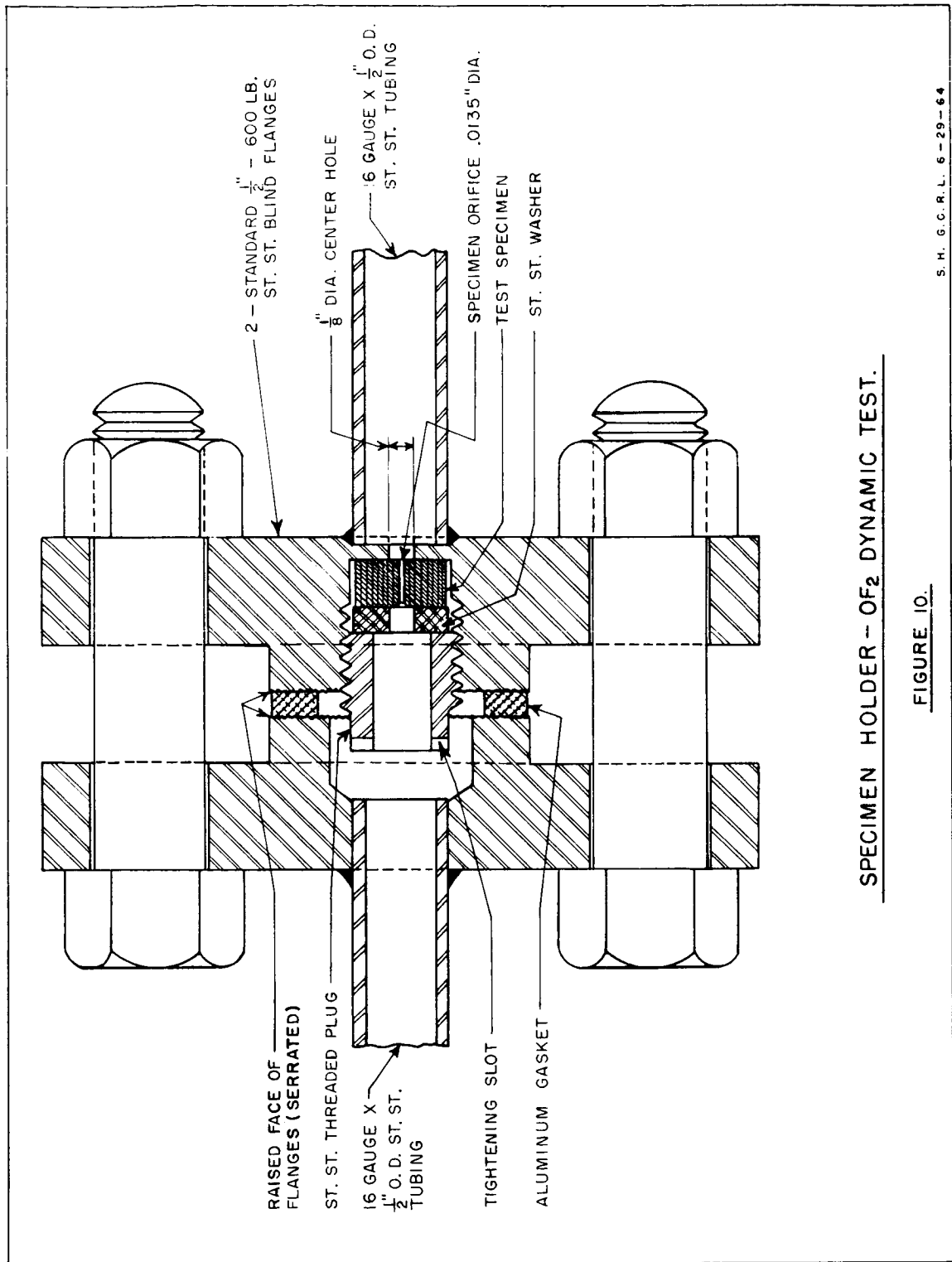
Instron Cryogenic Adapter
FIGURE 8



LIQUID OF₂ DYNAMIC TEST SETUP

FIGURE 9.

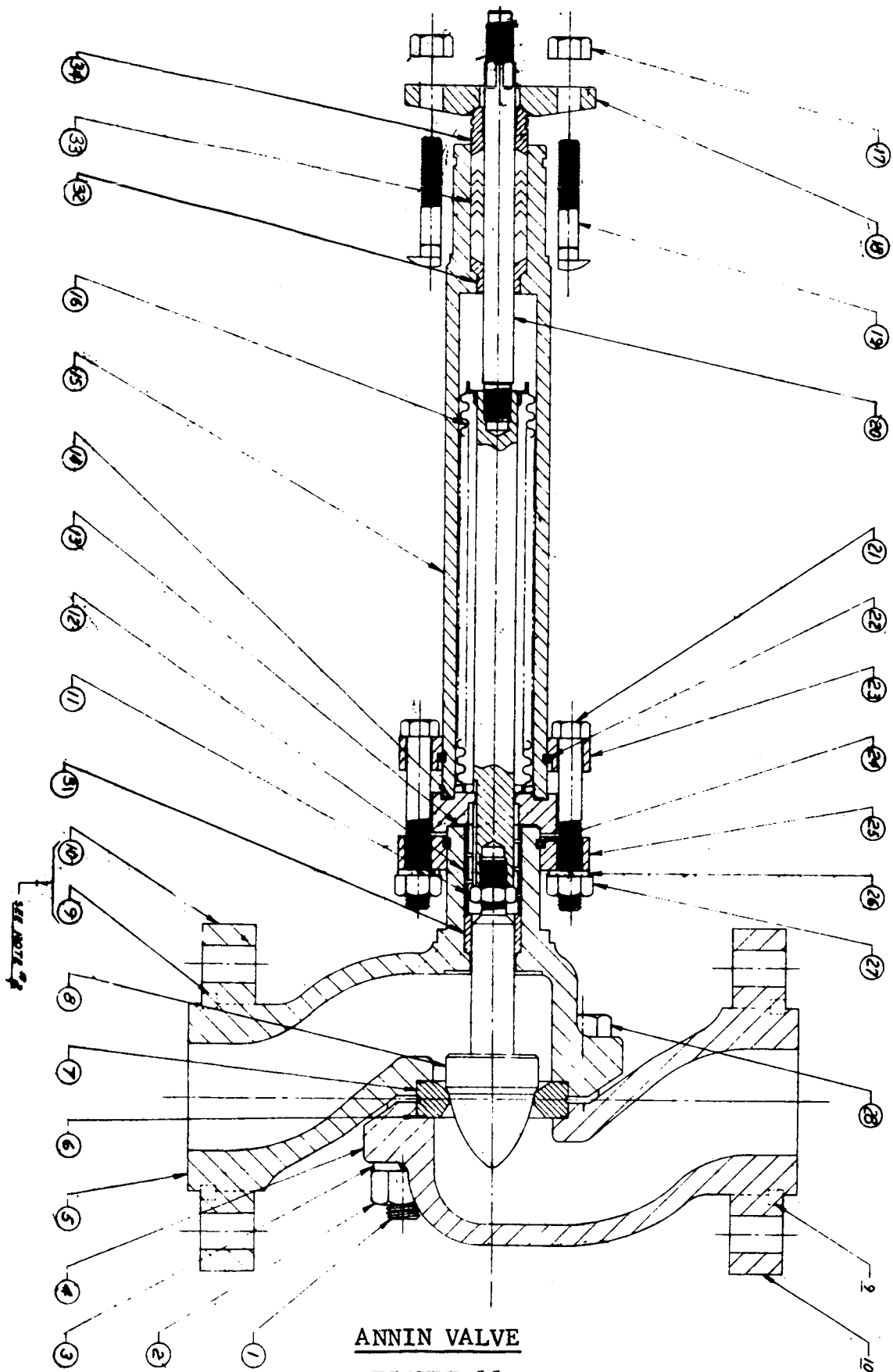
S.H.G.C.R.L. 7-1-64



SPECIMEN HOLDER - OF₂ DYNAMIC TEST.

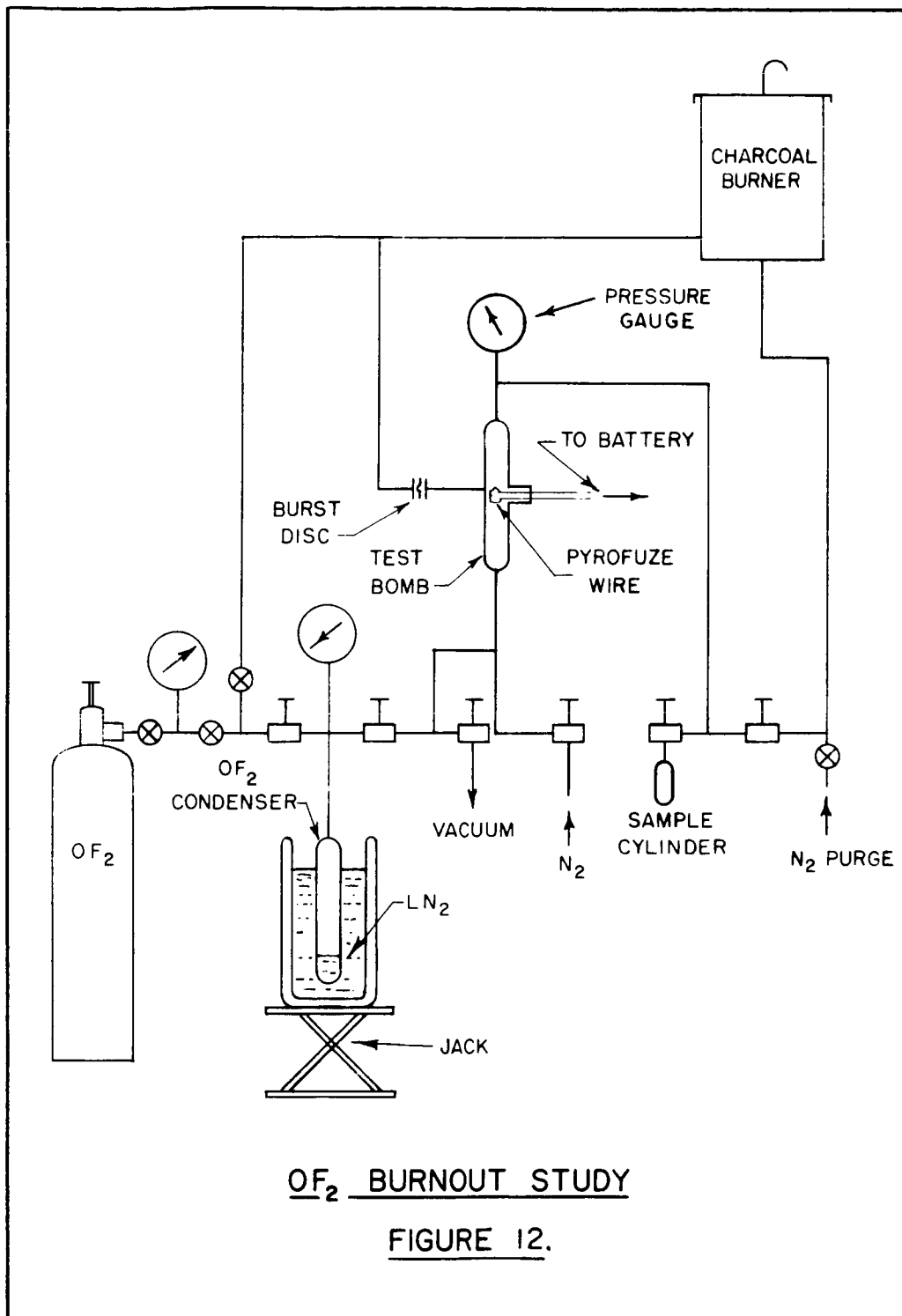
FIGURE 10.

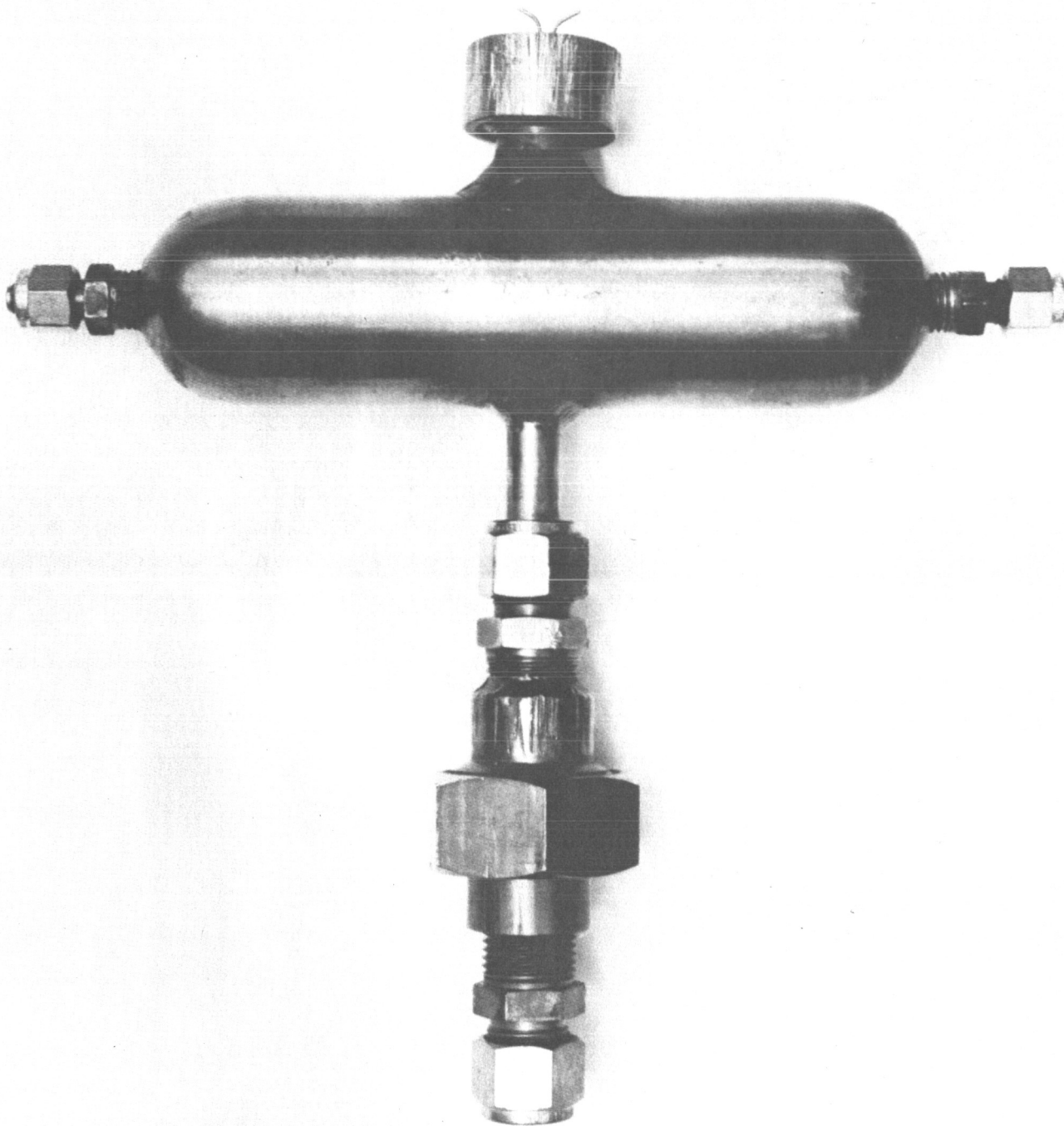
S. H. G. C. R. L. 6-29-64



ANNIN VALVE
FIGURE 11.

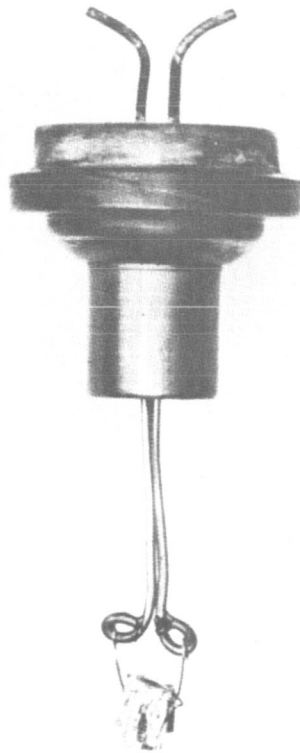
NOTE:
Refer to
TABLE 17





OF₂ Burnout Investigation, Reactor

FIGURE 13



OF₂ Burnout Investigation, Electrode Adapter
FIGURE 14

A P P E N D I X

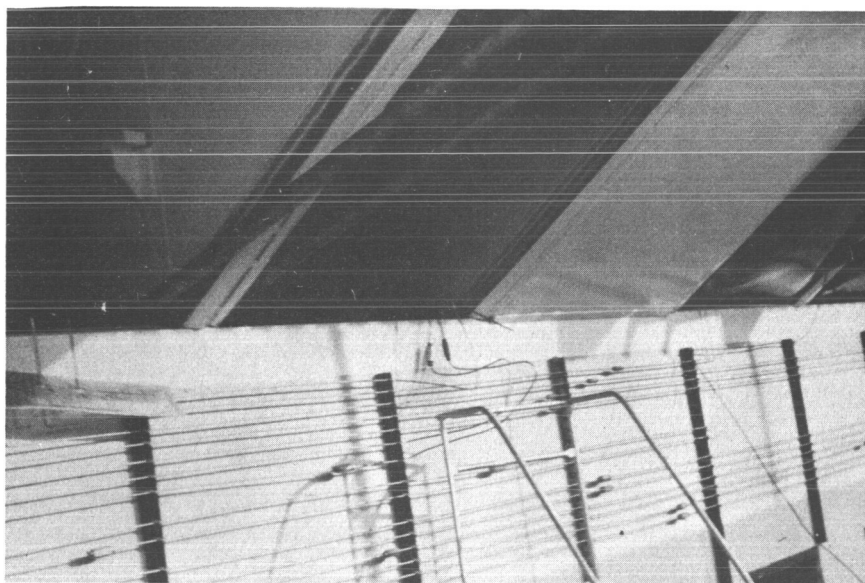


Exhibit A - Exterior of test cubicle.

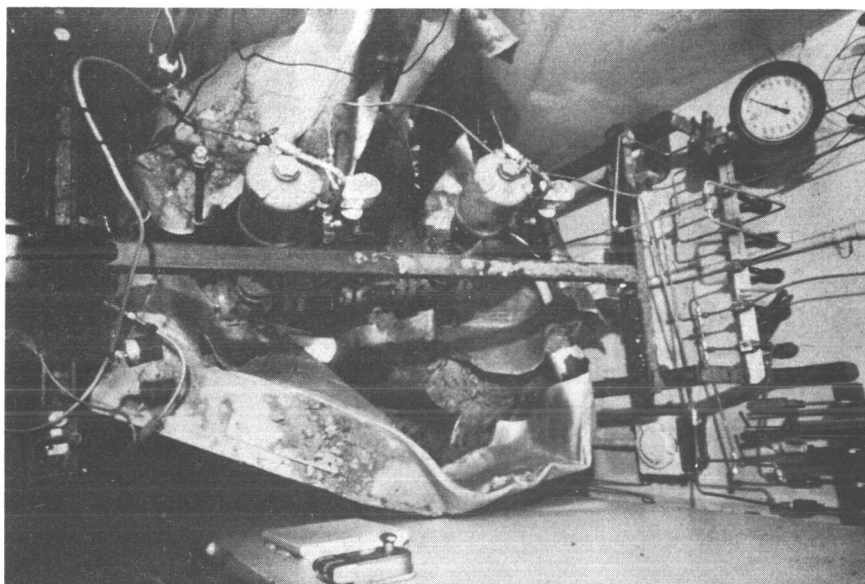


Exhibit B - Interior of test cubicle.



Exhibit C - Annin valve #1 and OF₂ reservoir.

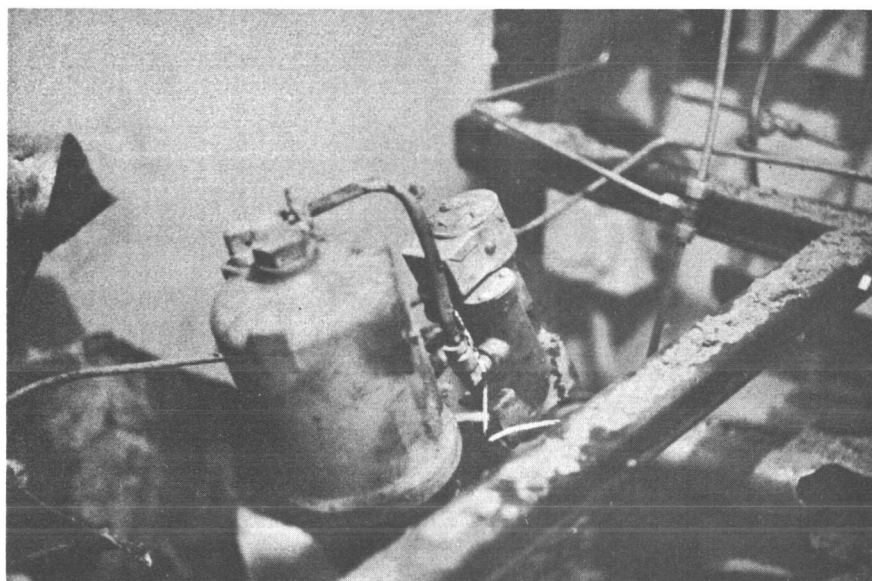


Exhibit D - Annin valve #1 dome motor.

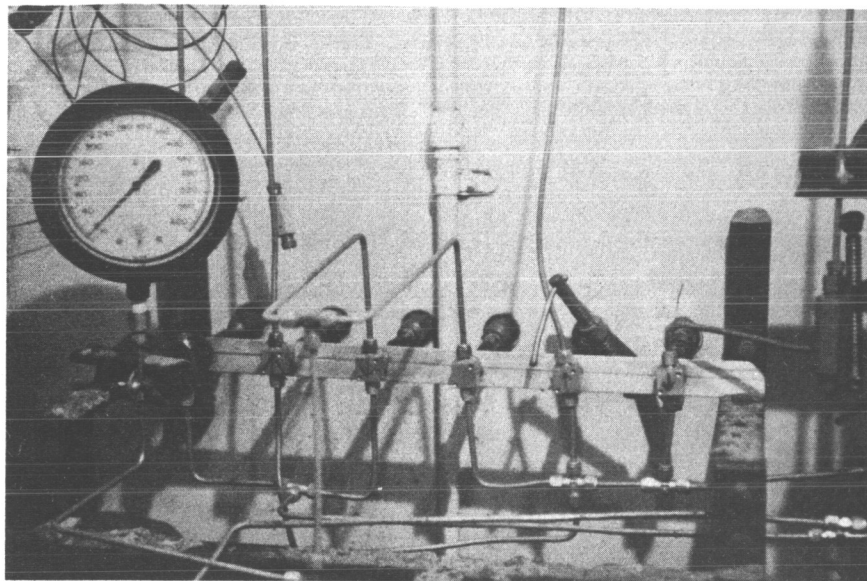


Exhibit E - Remote control valves and feed lines.

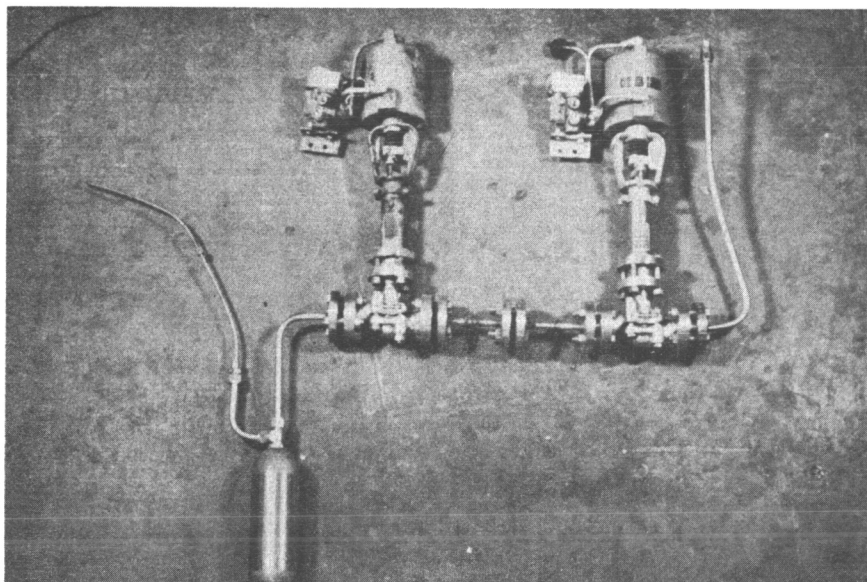


Exhibit F - Dynamic test set-up.

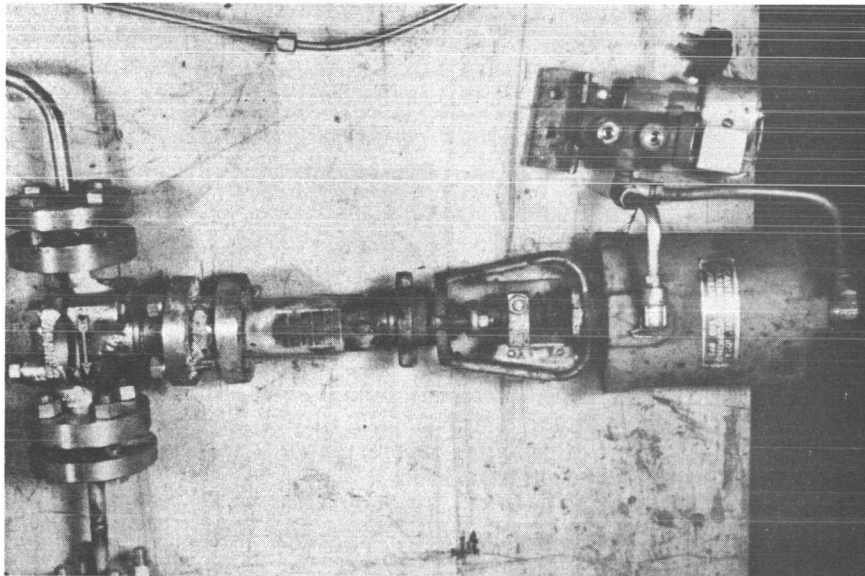


Exhibit G - Annin valve #1.

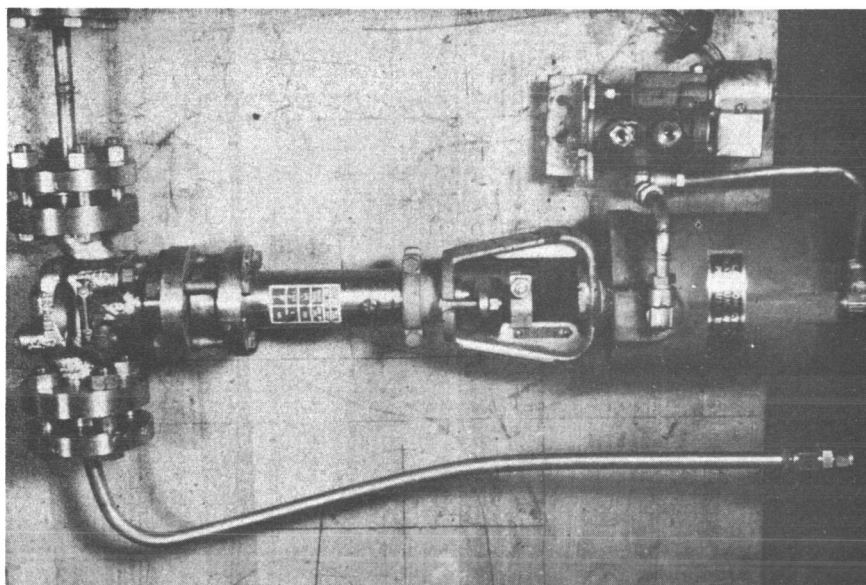


Exhibit H - Annin valve #2.

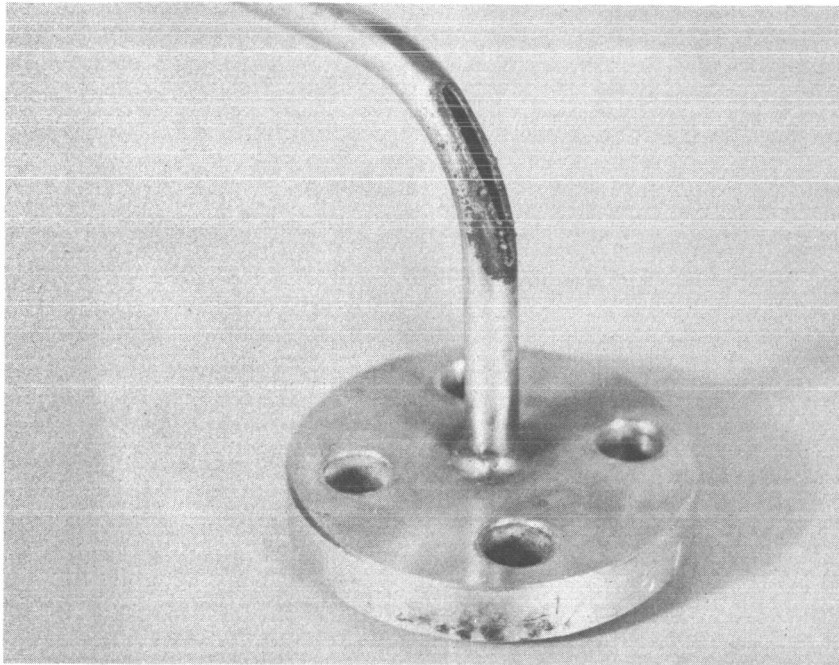


Exhibit I - Burned out elbow.

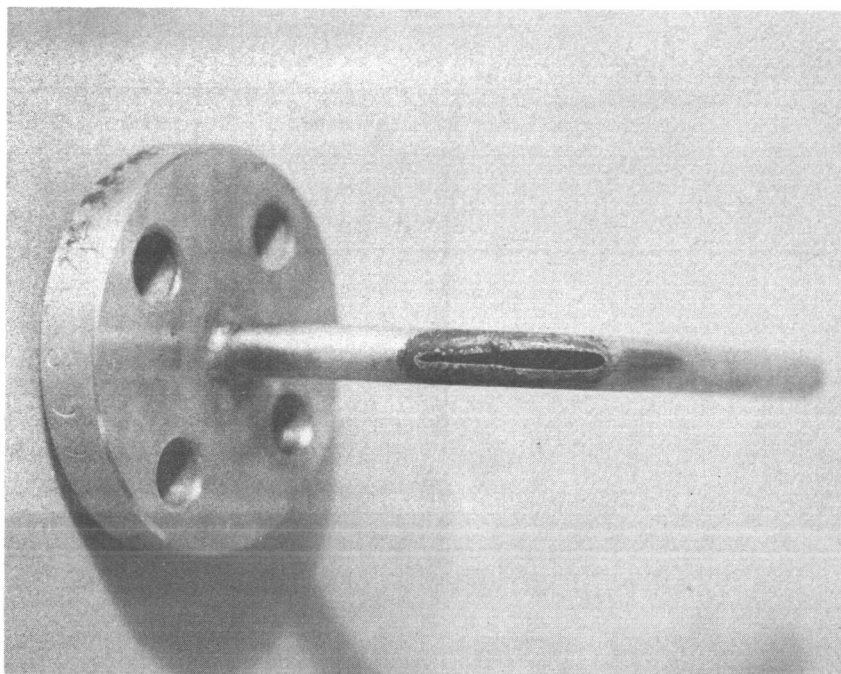


Exhibit J - Burned out elbow.

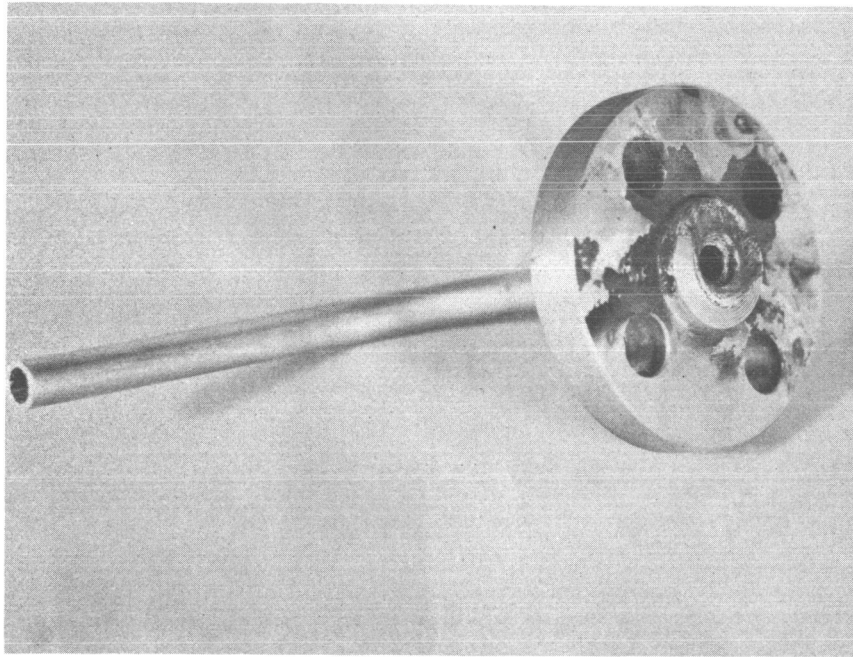


Exhibit K - Flange adjacent to Annin valve #1 inlet flange.

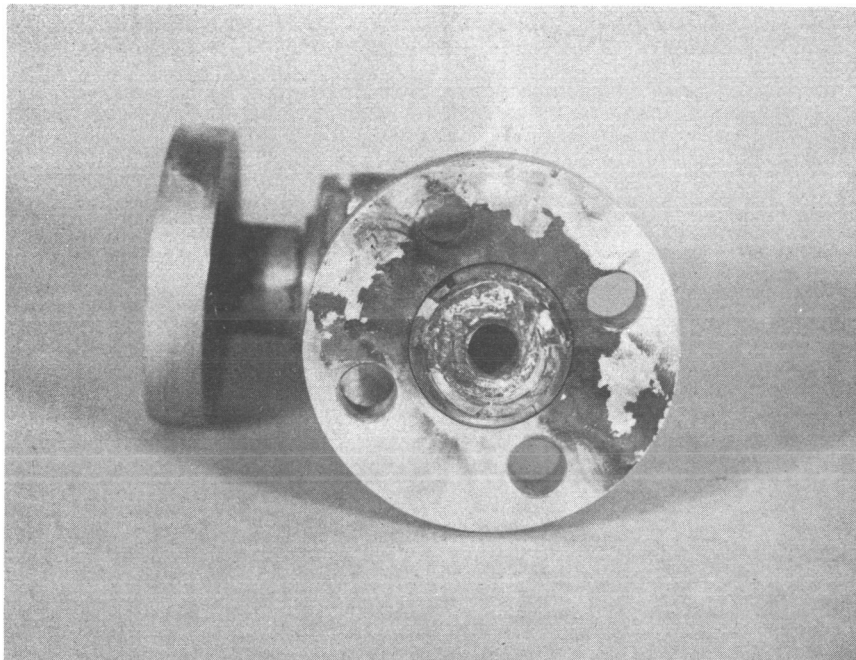


Exhibit L - Inlet flange Annin valve #1.

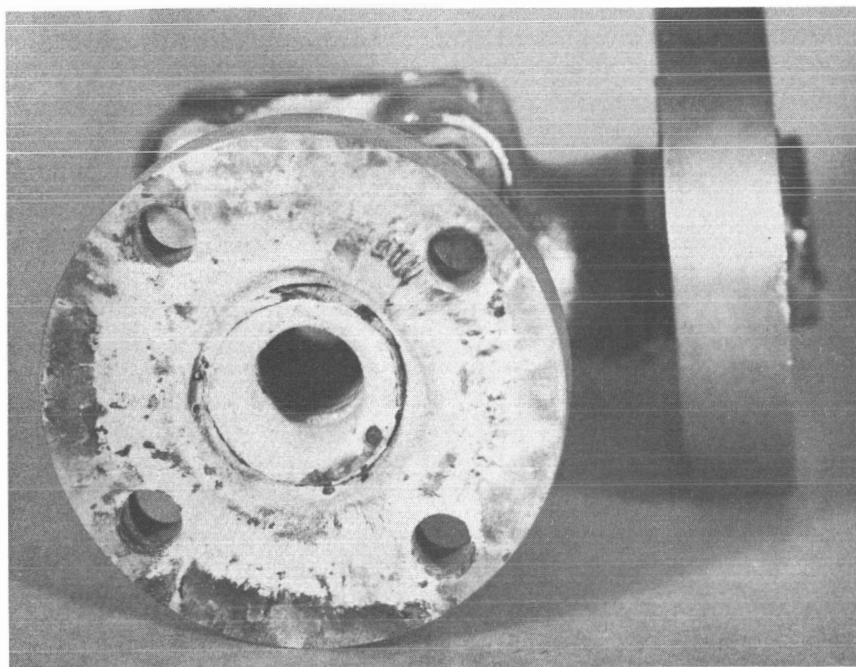


Exhibit M - Lower flange Annin valve body.

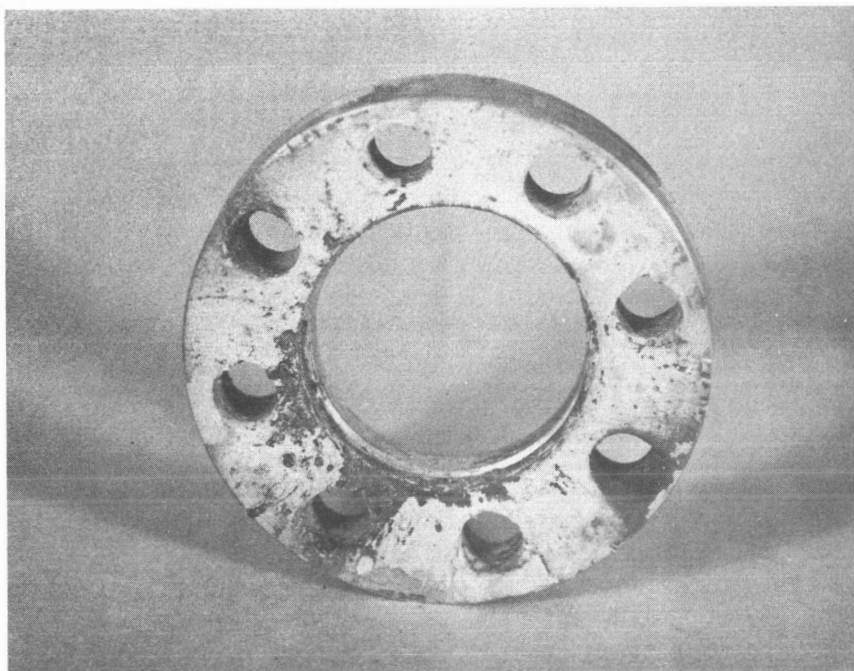


Exhibit N - Upper flange Annin valve body.

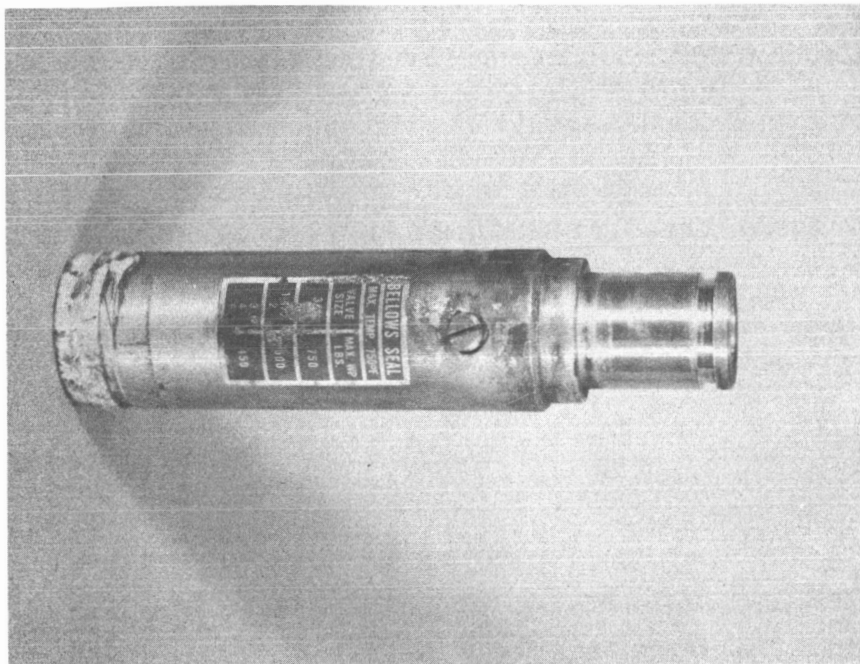


Exhibit O - Annin valve #1 body extension.

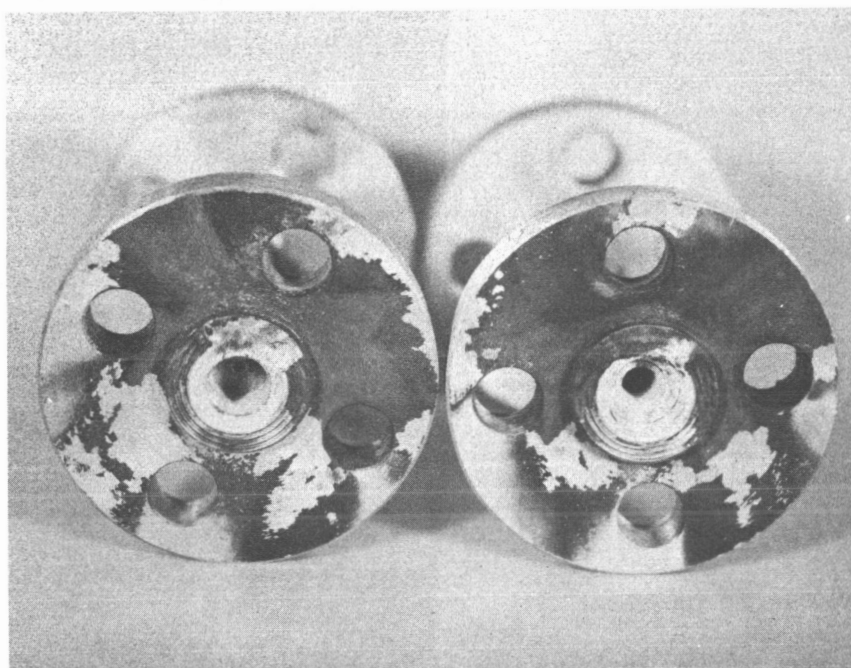


Exhibit P - Specimen holder, outlet side on the left.

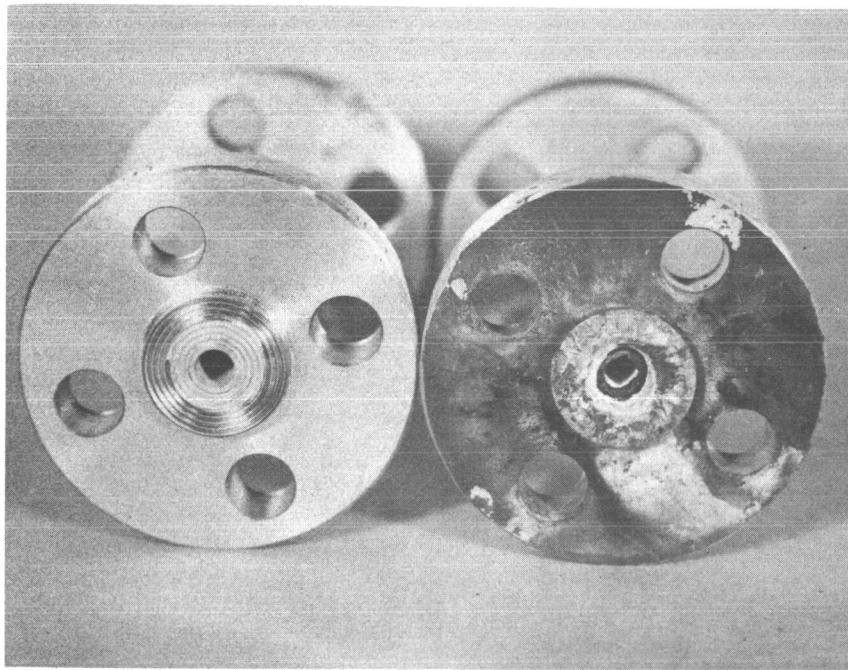


Exhibit Q - R to L. Mating flange to Annin valve exit, and first flange downstream from specimen holder.

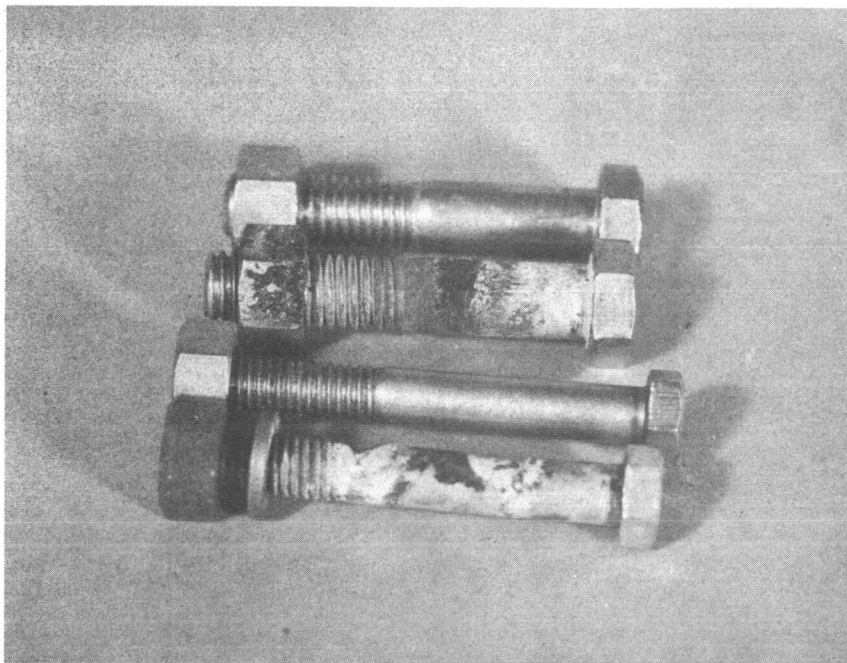


Exhibit R - Top - new and used flange bolts.
Bottom - new and used extension mounting bolts.

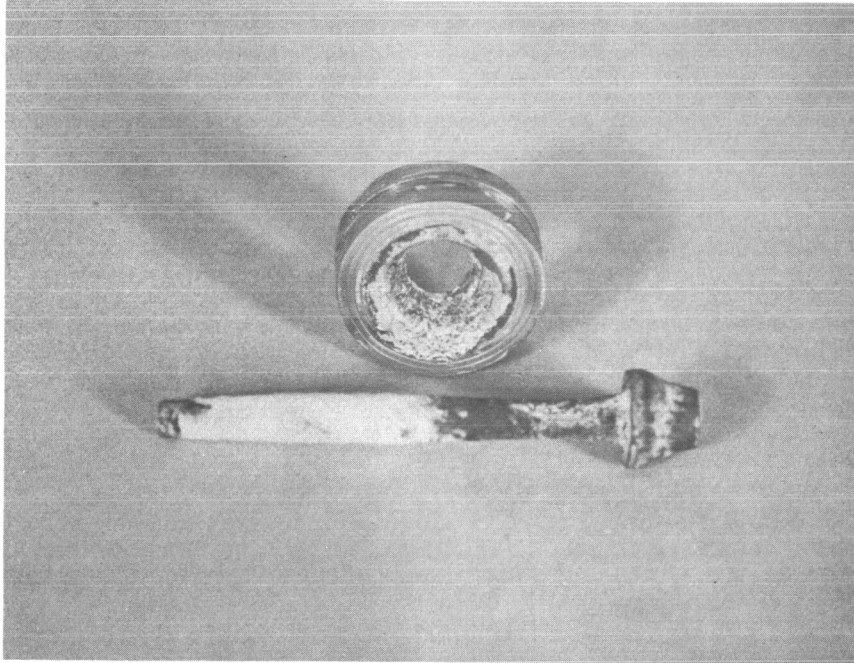


Exhibit S - Valve stem and seat from Annin #1.

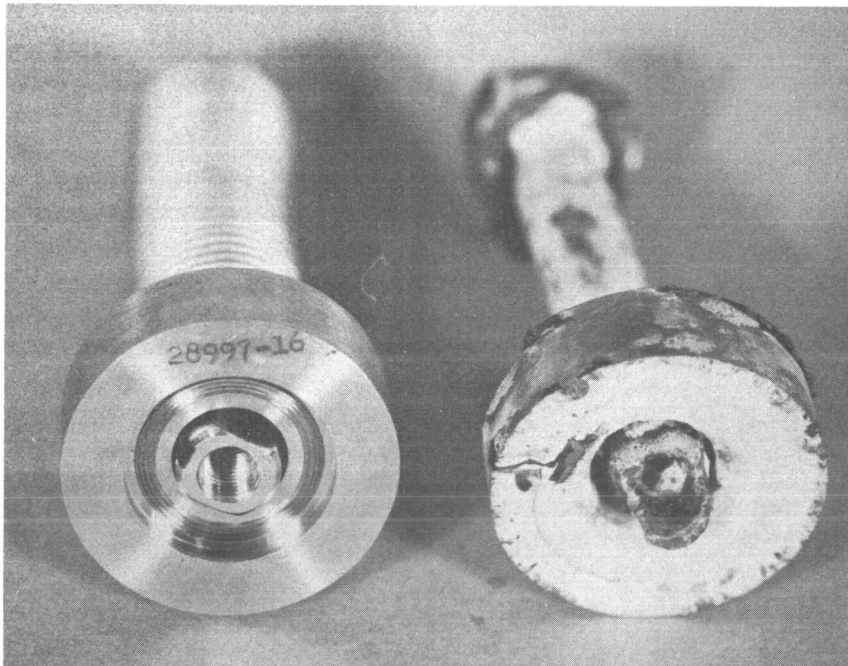


Exhibit T - End view bellows assembly.

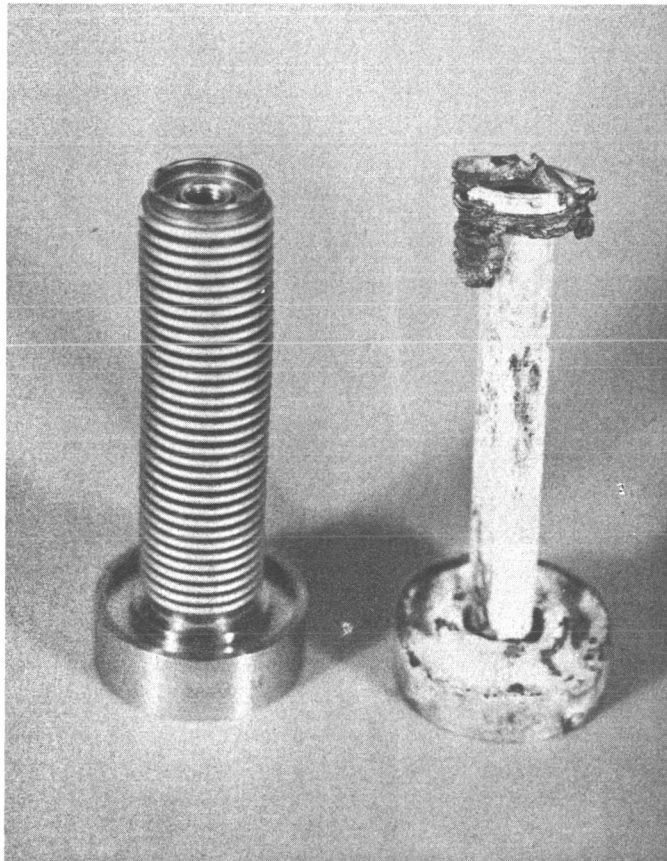


Exhibit U - Side view bellows assembly.

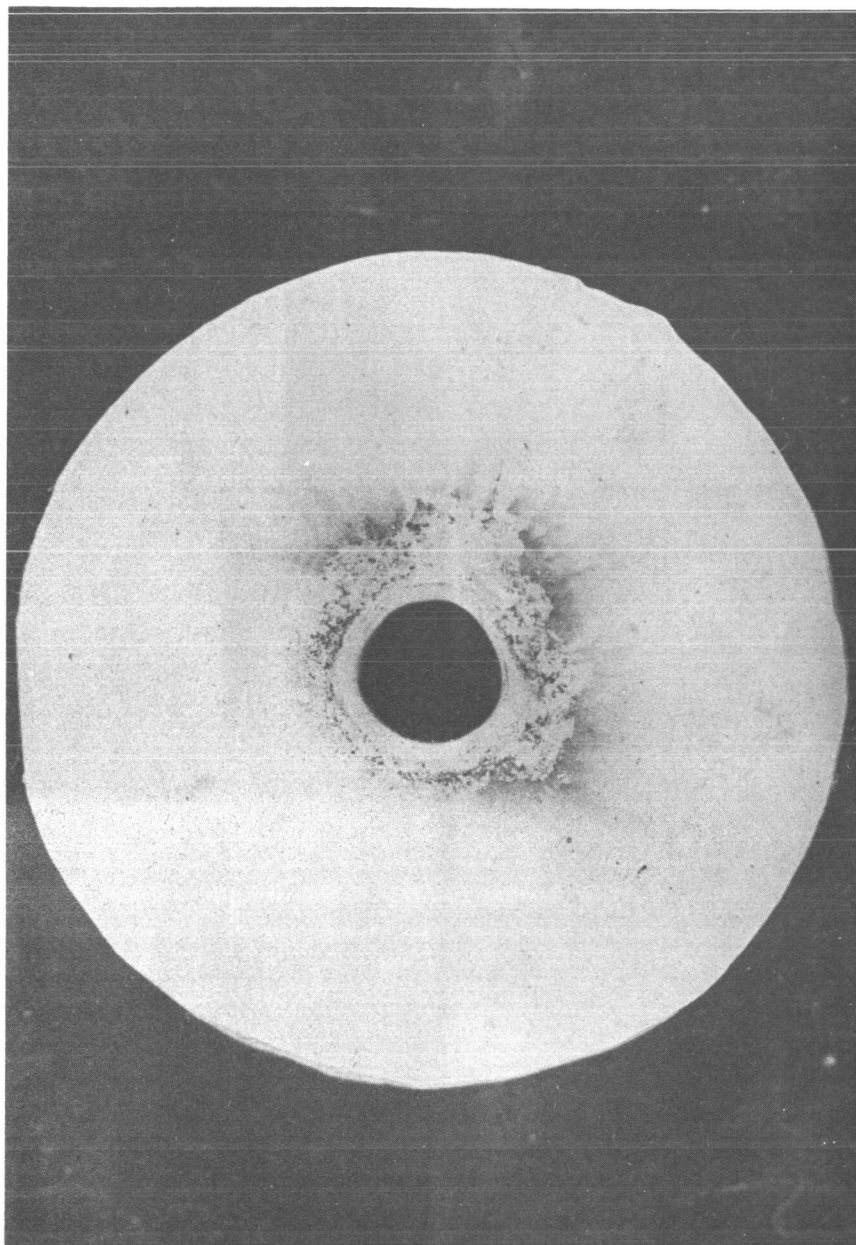


Exhibit V - Test specimen, inlet side.

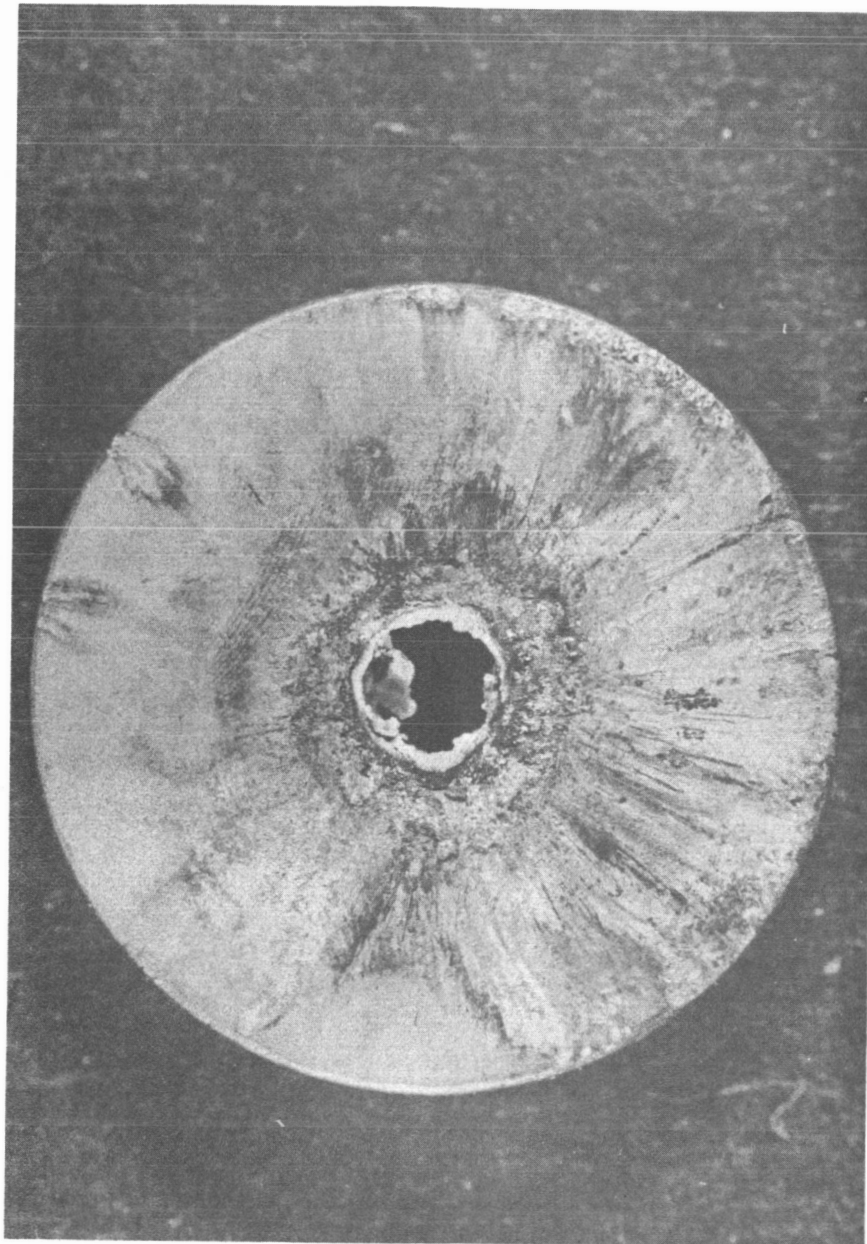


Exhibit W - Specimen thrust ring, outlet side.